Theoretical and Empirical Modeling of Flow, Strength, Leaching and Micro-Structural Characteristics of V Shaped Porous Ceramic Water Filters

DISSERTATION

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By

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Abstract

Clay based ceramic materials have been used in various parts of the world as a medium to purify and store water at points of use. United Nations and several other non governmental agencies are distributing these sustainable clay ceramic filters with embedded organic or carbonaceous materials to areas with scarcity of potable water. For example, rural areas in developing low income economies, disaster relief operation during flooding and many more on the list.

Several studies have taken place on these clay ceramic filters. There is a large amount of literature documented on the flow behavior through these clay ceramic filters, field studies on microbial removal effectiveness, structural damage of filters while in use and during transport, people/societies satisfaction and health impact of using these ceramic filters.

Here gravity operated frustum shaped clay ceramic filter have been manufactured using distinct volume ratios of clay and sawdust (organic material). This study has been done in order to individually assess the material parameter influencing flow, leaching and strength of these clay composite filters.

Reduction in flow is seen with time and filtration events imposing the need for scrubbing/cleaning of these filters. Similarly with fractional changes in embedded organic / carbonaceous material, there is a change in micro-structural and mechanical properties. Micro-structural, flow and mechanical characteristics change geo-spatially due to non availability of similar raw material constituents at different locations around the globe.

Functional challenges such as water contaminants, material composition are not the same everywhere. In this situation it is a must to investigate the stochastic effects on micro-structural, filtration flow/percolation and mechanical properties of these ceramic clay materials.

The cumulative flow has been modeled using a multi-parameter stochastic multiplicative model with time and volume fraction of the organic raw material used in the initial manufacture of the filter. The asymptotic regimes of cumulative discharge have been mathematically simulated using a predictor transformation for each of the different types of samples discussed. Material studies also confirmed a linear relationship between porosity and volumetric content of organic material used in the manufacture of these clay ceramics.

Mechanical properties of materials depend on the micro-structural features, compositional ratios as well as loading rates which they bear. Tensile fracture toughness development is found to follow a multiplicative behavior with respect to predictor variables like amount of organic material used for manufacturing, micro-structural porosity and applied force. Plastic flow during manufacture is found to influence mechanical properties in clay ceramics.

The alkalinity of the filtrate and its changes with time and material has been studied. Stochastic multivariate regression analysis on the principles of non equilibrium processes with respect to flow taking place through the filters has been used to provide extension to regression hypothesis proposed by Onasager for aqueous solution and porous flow mechanics. It was found that transport processes were dependent on each other but they also varied with time without losing their interdependence.

It is to be noted that microbial transport though these micro-nano porous ceramic filters are negligible. These filters showed similar microbial removal as confirmed by different authors earlier. This phenomenon may help in reduction of health problems due to drinking water. So in order to test these filters in an economically low income society setting, these filters were introduced in Eweje Village, Ogun State, Nigeria. A nine month survey was conducted with an objective to find the independent variables which may influence health in areas where these filter are being utilized. It was found that time of filter usage, traditional medicinal knowledge/medical facility access and money were the top influencers of health in Eweje Village, Ogun State, Nigeria.

Dedication

This document is dedicated to my parents Parameshwara Krishna Plappally and Thankom Plappally, teachers from Kerala School, teachers through by bachelors, advisors at Indian Institute of Technology, CIMMYT-RWC & University of Alabama, Tuscaloosa and my current advisors at the Ohio State University.

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Publications

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Fields of Study

Major Field: Food Agricultural and Biological Engineering Program

Studies in: Flow Mechanics, Structural Mechanics, Material Science, Thermodynamics, Probabilistic Methods in Engineering and Hydrology.

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Chapter 1: Present Scenario of Water Purification at Point Of Use in the World: Review of parameters and their influences

Abstract

In recent years there has been a looming concern over availability of water, especially potable and pure water. Societal influences, water scarcity and purity of water are closely related. Income, educational awareness, accessibility and sustainability have become the major paradigms influencing water purification technologies in developing countries. Chemical reagents, structurally modified local natural materials, organic constituents and manual skills are the key factors affecting sustainable low cost technologies. Clay ceramic filter is one such example. This chapter reviews water contaminants, availability of water and current technologies for water purification at point of use. It also discusses on why ceramic filters have been studied and how these studies will help ceramic filter manufacturability, functional feasibility and sustainability.

1.1 Introduction

Natural waterways facilitated the spread of human beings to various landscapes and societies to strive. It is important to look at the history of point of use water treatment. Human habitats were dotted near sources of fresh water. For example, Egyptians settled along the Nile, Harappa Civilization along the Indus, the Incas around Lake Titicaca in Peru and similarly for other civilizations. So the purity of water from these sources of fresh water becomes important for sustenance of life. It is to be noted that these human habitats were also the sources of pollution of fresh water [Stefferud, 1955].

Purification of water by solar disinfection and boiling water at high temperatures has been dated back before 2000 BC [Srikanthamurthy, 2004, Van

Loon, 2003]. Material science (Dhatu Shastr) was also found to be highly developed for water purification and medicinal enhancement. For example, water was kept in copper vessel for reducing blood pressures or for disinfection, properties of silver(Ag) as a bactericide, clay pots (for cooling water) and also addition of birthstone named Gomedaka into water [Srikanthamurthy, 2004, Van Loon, 2003].

Sterilization and storing of drinking water in vessels made or lined with materials such as silver and copper are prehistoric and also as recent as drinking water recycling in NASA space shuttle [OTS, 1996, Sagara, 2000].

In 1627, Sir Francis Bacon compiled the book titled "*A Natural History of Ten Centuries"* that was on water purification and desalination [OTS, 1996]. In 1685, Lucas Antonius Portius wrote on water purification with multiple sand filters in his book "S*oldier's Vade Mecum"* [OTS, 1996]. A proposal in France dating back to early 1700 considered that every household should have a water filtration system with sand and a large vessel for collecting rainwater [OTS, 1996]. Hyatt filters (1880), Alum and Retention Soil Filters (1885) were introduced after the cholera outbreak in New York in 1840 [Diodorus, 1939]. Lime soda softening process of water was introduced in 1903 and chlorination in 1908, after more than 1000 deaths were reported due to typhoid in Philadelphia in 1900 [Diodorus, 1939]. Granular activated carbon for water purification was introduced in 1930 [EPA, 2000]. Disinfection byproducts (DBP) were later discovered in 1974 which alerted the people to problems caused by excessive use of chlorine. This helped the development of membrane technology and filtration. Reverse osmosis and electro-magnetic radiation technologies were not researched to purify water until late 1990s [Crittenden et al, 2005, Marshall, 2000]. It is interesting to note that discoveries and methods followed after the disease outbreak. These discoveries are basically materials related and depended on material properties and their interaction kinetics with the aqueous phase.

1.2 Current Scenario of Availability of Water

It is very important to know the percentage of the population we have to cater with improved techniques to purify the fresh water in economically backward countries. From the Fig 1.1 it is very clear that more than 50% of rural areas on Earth are still having scarcity of potable water. It is also known that more than half of the rural population around the world is below poverty line [UN, 2006].

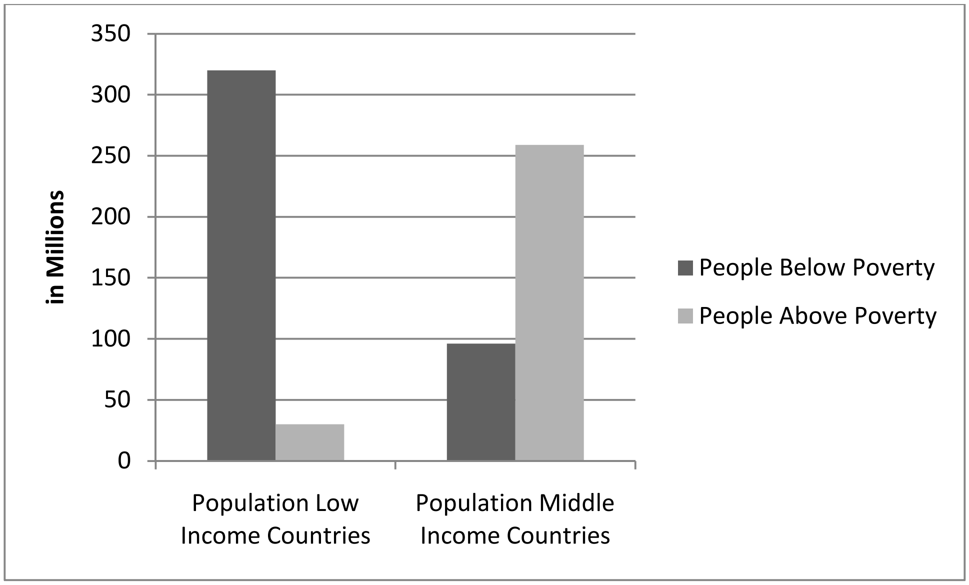

Figure 1.1: Population without access to potable water around the globe [UN, 2008].

North America has better economic condition, hence advanced research and development has made it possible for people to access quality potable water which is clear from Table 1.1[Gadgil, 1998]. It is also to be mentioned that 1990 -2000 was a decade of rapid progress in African nations on this matter, but UN-Miilliniem development Goals (MDG) for 2015 will never be met at this pace [WHO/UNICEF 2004].

Populations in millions across earth without access to potable water as of 2002 [WHO/UNICEF 04]	
Latin America & Caribbean	60
Sub-Saharan Africa	288
Northern Africa	15
Developed Regions	15
Eurasia	20
Oceania	3
Western Asia	23
South-Eastern Asia	115
South Asia	234
Eastern Asia	303

Table.1.1:Geospatial demographics of the scarcity of potable water in 2002.

This is also confirmed by data of the WHO/UNICEF 2004 meeting as shown above in Table 1.1, which shows the population without access to potable water in 2002. The rural society has been taken into account as the majority of the developing nations are agrarian societies.

1.3 Contaminants and their Current Removal Technologies

Drinking water sources such as rivers, lakes etc, have been polluted with inorganic fertilizers, pharmaceutical compounds, organic compounds from industries, nuclear waste spills, chemicals used for waste water treatment purposes, biological contaminants (human and animal feces) and many more to list. So after looking into this long list of impurities, it is clear that besides anthropogenic sources geo spatially distributed point sources are also contributors to water pollution.

1.3.1 Geological Contamination of Drinking Water and its removal

Studies from Asia (Taiwan [Chiou,et al. 2001], Japan [Tsuda, et al. 1995], and Inner Mongolia, China [Tucker, et al. 2001]) and Latin America (Chile [Smith et al. 1995; Ferreccio et al, 2000], Argentina [Hopenhayn-Rich et al, 1996], and Mexico

[Morales et al, 2001]) indicated that arsenic in drinking water could cause skin, bladder, liver, or lung cancer [Brown et al., 1989, Lamm, et al. 2004].

Arsenic leaches into drinking water (135μg/L) from rocks and soil in Nepal. Sarkar et al., confirmed that in all arsenic contaminated groundwater, dissolved Iron is also significantly present and often at concentrations greater than 2.0 mg/L. Groundwater in Nepal, India (Bihar and West Bengal), Bangladesh,Vietnam, Cambodia, and Mexico also has the above mentioned feature [Sarkar et al, 2008].

Point of Use (POU) water filters with appended arsenic removal media comprising of columns of iron nails, cloth (sari), fine and coarse sand leveled one over the other was used successfully in Nepal [Sagara, 2000, Ngai, 2002, Hillie, et al, 2009].

There are several other POU technologies and chemical to remove arsenic from drinking water [Sharmin, 2001]. They are iron coated sand [Joshi et al, 1996], activated seawater-neutralized red mud, referred to as activated Bauxsol [Genc-Fuhrman et al, 2004], mixture of powdered iron, sulphur and hydrogen peroxide, sunlight or ultraviolet rays, zero-valent iron appended with limestone [Kanel et al, 2006, Lackovic et al, 2000, Welch et al, 1981], slag from steel furnace, zeolite and ion exchange resin (together form permeable reactive barriers for arsenic removal), coagulation [EPA, 2000], iron oxide coated sand and manganese oxide coated sand tested by Thirunavukkarasu et al. in 2002, ENPHO and MIT made Kanchan MIT Filter [Ngai, 2002]. Arsenic is also removed using ceramics made of clay (Kolshi Filter, Nepal) with high Fe content. There has been a trend recently to move back to old technology of taking water from dug wells and sand filtering or auto-attenuation[EPA, 2000, Sagara, 2000, Hwang , 2003, Joya et al, 2006].

In Bangladesh, an experiment was conducted based on sand filter , a chemical mixture of approximately 1.5 g of industrial grade ferric sulfate ($Fe_2(SO4)_3$) and 0.5 g of calcium hypochlorite ($Ca(OCl)_2$), and fabric clothing. Here both sand and chemical mixture was treated with each other to prepare the removal column [Cheng, et al, 2004]. Humic acid which is a derivative of cellulose has been found to give advantages while filtering with zero-valent iron or iron nail type objects [Giassudin et al, 2007 and Gu et al, 2005].

Different methods such as use of low density current to mobilize arsenic ions (electro-deposition or co-precipitation or ion exchange) and phyto-degradation also contribute to arsenic removal [EPA, 2000].

Organ damage (kidney, liver and colon) and cancerous growth was reported in human beings due to drinking water polluted with metallic materials such as chromium, barium, lead, mercury and selenium [Dorman, 1996]. Silver in excess of 0.05 mg/L may cause Argyia [Ma et al, 2007].

1.3.2 Anthropogenic Contamination of Drinking Water and it removal

As per the WHO (1996), about 96.5 % water on the Earth is in oceans, seas and bays; 1.74 % is from glaciers; 1.7% is groundwater; 0.76% is fresh water and rest is saline, soil moisture and permafrost. The ground and freshwater occur in very low quantities as compared to the total water available. Fertilizers, basically nitrate based compounds have been added to the fields to provide plenty of nitrogen for the plants. Nitrate leaching through the soil can pollute potable water sources. Nitrate goes into human system and get converted to nitrite causing methemoglobinemia in infants and colon cancer in adults [Fewtrell, 2004, Yang et al. 2007].

Pesticides, insecticides and rodenticides are other manmade chemicals that reach fresh water sources when ingested they causes cholinesterase inhibition; reproductive and immune system effects, cancer and damage to eyes, kidney and liver [Dorman, 1996].

1.3.3 Microbial Contamination and its Removal

The other major pollutant materials are biocontaminants that are responsible for water borne diseases. The animal and human excreta as well as biological wastes carry pathogens including bacteria (e.g. E. coli, vibrio cholerae, Campylobacter jejuni, C. coli etc), viruses (Adenovirus, Norwalk, Rotavirus etc), protozoa (E.g Entamoeba histolica, Giardia intestinalis, Cryptosporidium parvum, etc), parasites or helminths (Trematodes, Cestodes, Nematodes species) [Clasen et al, 2006].

The microbiological prespective of water was in focus from the early 1850s. Important preliminary correlation between potable water quality and microbial activity was developed by Hassell [Rafter, 1892]. First cases of health deterioration due to drinking water contaminated with bacteria were published in 1850s [Snow, 1855, Budd, 1857]. It is also very important to note that microbial deactivation experiments with the help of sunlight were also published in the same period [Downes et al, 1887]. Removal and inactivation of microbes in drinking water supply has been published in various research publications around the world. Some of the earlier ones are consolidated in the table below.

Microbe	Treatment Process	Reference
Virus- Viral plaque forming units	Coagulation, Sedimentation, Flocculation	[Clarke et al, 1959], [Gerba, 1984]
Bacteria-E. Coli, Salmonella Typhi, Psuedomonas pyocyanea	Slow Sand Filtration, Flocculation	[Wallis et al, 1965], [Baker, 1949], [Butterfield, 1948]
Actinomycetes	Chemical Coagulation, Solar Disinfection	[Burmen , 1973, Downes et al 1887, Niemi, et al, 1982]
Fungi	Chemical Coagulation, Disinfection	[Burmen , 1973, Niemi, et al, 1982]
Yeast	Chemical coagulation	[Niemi, et al, 1982]
Protozoa	Coagulation, Flocculation, Sedimentation. Filtration	[Sobsey, 1983]

Table 1.2: Some review publications related to microbial removal processes.

Theodor Escherich in 1885 discovered a bacterium commune (E. coli) in the stool of infants and found that it easily grew in lab environment [Friedmann, 2006]. The first book on microbiology of potable water “Guide to the Microscopical Examination of Drinking Water” came into print in 1875 [Olson et al, 1984]. American Public Health Association brought the first edition of “Standard Methods for the Examination of Water and Waste Water, in 1904 [Olson et al, 1984].

Table 1.3 shows the prominent water related diseases causing widespread mortality.

Categories	Cause	Diseases
Waterborne	Intake of water polluted by human and animal feces	Cholera, Typhoid, Amoebic and Bacilliary dysentry, Trachoma
Water based/related/ unsecure ponding/collections	Host organism parasites living and insects breeding in water	Parasitic: Dracunculiasis, Schistosomiasis etc. Insects: Malaraia, Dengue, Yellow fever, etc.
Toxic / Poisoning	Eutrophication of surface water bodies	Gastro-intestinal or Hepatic illness

Table 1.3: Water related diseases and their causes [UN-Water, 2006, WHO, 1996].

Other than the above mentioned technologies, materials such as bleach, PUR (ingredients include ferric sulfate, bentonite, sodium carbonate, chitosan, polyacrylamide, potassium permanganate, and calcium hypochlorite) had been tested againt microbial contamination and were promising in Guatemala, Kenya, South Africa, Pakistan, Bangladesh and Philipines[Souter et al.2003, Reller et al. 2003, Crump, et al. 2005]. It was found effective in removing microbial contaminants and also tested for arsenic removal [Souter et al.2003, Reller et al. 2003, Crump, et al. 2005].

Table 1.4: The different POU water purification processes, their effectiveness , quality and sustainability.

A1- Chlorites and Coagulants, A2 – for Chlorine, F1- Ceramic filter, F2- Sand Filters, U1-SODIS PETs.

Please Note: [Here references are given in a different format]

Treatment	Chlorination	UV	Ozone	Heating	Filtration
1a. Microbiological removal effectiveness :	Bacteria-A1[1] Log Reduction ~7-9 Virus-A1 Log Reduction ~2-6 Protozoa-A1 Log reduction ~ 3-5	Bacteria – U1[1] Log Reduction ~ 3-5.5 Virus – U1 Log Reduction ~ 2-4 Protozoa- U1 Log Reduction ~ 1-3	99.99 % effective in removing microbes [2,3]	Bacterial pores and cysts do not survive heating [4] Viruses become inactive but are not killed by boiling.[5]	Bacteria – F1[1] Log reduction ~ 2-6 Virus – F1 Log reduction ~ 0.5-4 Protozoa – F1 Log Reduction ~ 4-6
1b. Microbiological removal effectiveness	Bacteria – A2 [1] Log reduction~ 3-6+ Virus – A2 Log reduction ~ 3-6+ Protozoa- A2 Log reduction ~ 3-5				Bacteria – F2[1] Log Reduction~ 1-3 Virus – F2 Log Reduction~ 0.5-3 Protozoa – F2 Log Reduction ~2-4
2. Ability to meet USEPA's Drinking Water Standards	Trihalomethane concentration should be less than 0.1 mg/L. This produces other chemicals such as aceto nitriles. [6] EPA goal : MRLDG 4 (mg/L)[2] [6] EPA goal for haloacetic acids: dichloroacetic acid (zero); trichloroacetic acid is <(0.3 mg/L) [7]	It is able to kill major bacteria from water but is not good enough in reducing E. coli and other thermo-philic micro organisms. [14] EPA goal :2 LRV for bacteria -Surface area and Intensity based	Better than Cl and UV in meeting the EPA standards with much wider microbial disinfection.[2,7] Results are quantity specific [6]	Water with impurities other than micro organism should be controlled (metallic compounds calcium and magnesium carbonate, which form scale) and looked at to prevent increase in concentration of that substance due to evaporation. [15]	EPA routine test has < 10 colonies per mL 3-4 LRV for bacteria and Virus. [13] ***Continued Table 1.4*** Continued Table 1.4

Continued Table 1.4

Continued Table 1.4

Treatment	Chlorination	UV	Ozone	Heating	Filtration
EPA rules effectiveness		EPA approves maximum contaminant level (**MCL**) of zero for total coliforms in a 100 mL sample of drinking water [3,8,13]	3 Log reduction for bacteria and 4 Log reduction for virus,ie a 99.9 % and 99.99% removal/inactivation correspondingly [3,7,9]		
3. Initial Cost :	Chlorine is supplied as concentrated liquid or sachets Cost of production is ~$ 3800 /mgd PUR costs ~$4230/mgd [2]	PET bottles are usually discarded bottles. Low Cost [1]	~$30000/mgd is the cost of production of ozone for purification purposes. The disinfection demand high oxidation hence consumption rate is high [2]	Energy Consumption is high [9,15] Constant heat supply is required.	High cost for material and chemical used . Ceramic Filter ~$8-10 per unit[1] Sand Filters ~ 100 $ per unit[1]
4. Operation and Maintenance Cost	Production of the chlorine tablets or sachets go on varying from $0.003/L > $0.01/L $0.08 to $0.20 per pound of liquid chlorine **[8]** $0.60 to $1.00 per pound Cl_2 **[8]**	Low [7,14]	Due to high instability of ozone and corrosion, the equipment cost and maintenance cost goes high	With increasing energy prices the cost may go up.	Long term replacement or exchange required. [1,10]

Continued Table 1.4

Continued Table 1.4

Treatment	Chlorination	UV	Ozone	Heating	Filtration
5. Time required for treatment	Requires a constant supply	Long 6-8 hours of constant sunshine [14]	Due to ozone instability in water the rate of reaction is very high.	5-10 minutes of boiling and for every 500m head above sea level, 1min extra should be added to the time of boiling. [11,5]	For various type of filtration technologies, the available time varied from around a minutes to couple of hours. [1]
6. Training Requirements:	Hypo-chlorination and chlorination equipments should be known to used and operator certified.[5, 12] Chlorine gas and hypochlorite are hazardous materials hence safety training and chemistry behind the usage and amounts should be known. [6]	Care to be taken while filling up the PETs , such that no other impurities get into the waters. As other impurities can influence UV biocide in a negative way [4]	99.99 % effective in removing microbes [2,3]	Bacterial pores and cysts do not survive heating [4] Viruses become inactive but are not killed by boiling.[5]	Training for manufacturing and application of chemical on filters. Dissemination programs. [1]
7. Other limitations (Disinfection by products) DBPs.	High corrosion, reactive to metals, prominent in slurry and scale formation, formation of haloacetonitriles, halo ketones, n- organo chloramines[6,7]. EPA – Tri halomethanes under DBP rule for stage 1 should be lower from 100µg/L to 80µg/L. MRDL for Chlorine 4 mg/L [2, 8, 7] For chlorine dioxide is 0.3mg/L [6,7]	Less quantity of water can be sterilized at a time. [1,13]	Very high cost, large quantity usage, ozonation DBPs is basically aldehydes, carboxylic acids, hydroxy ketones, di carbonyls, ketones and keto acids. Due to reaction with organic materials it also leads to formation of humic acid. [7] Bromate compound formation.[6]	No such DBPs, but if some salts are present their concentration in water increases with time due to evaporation of water. [12, 14, 15]	Structural anomalies cause breakage while in use. [1] Frequent replacement of disinfecting components such as activated carbon, iron coated and alumina Fe sand are to be discarded due to formation of harmful complexes. [1]

Source:

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1.4 Drinking Water Related Health Statistics around the World

The incidence of mortality rates due to drinking water related diarrhea decreased from around 5 million in 1980 to around 1.5 million in 2000 [Davis, 2008].
WHO suggested in 2008 that 10% of the diseases in this world can be reduced by improving and managing drinking water supply, sanitation , hygiene and water resources respectively [WHO, 1996].

Almost 1.4 million children aged less than 5 years die due to water related diseases of which 90% is due to diarrhea with a rate of deaths per quarter of a minute [Boschi-Pinto et al 2008].

1.5 Problem Description with Emphasis on Point of Use Treatment of Water

Potable water availability is a necessity in rural and economically backward regions around the world. Large centralized systems may not be good for serving dispersed unplanned rural areas, and hence a decentralized point of use household technology is found to be viable [Montgomery, 2007]. Improved drinking water sources were cited by the WHO/UNICEF in 2004, as namely household connections, public standpipe or hand pumps, borehole, protected dug well, protected natural spring and rainwater harvesting[WHO/UNICEF, 2004].

It is a reality that with improvement in science and technology the complexity of the compounds and microbiological knowledge of raw water has also gone up. But basic thought of purification remains the same. Water purification and filtration methods must be low in cost, socially and culturally acceptable and should be technologically feasible for the local environment. These methodologies should be easily accessible to the people in need around the globe [Hillie, et al, 2009].

Water scarcity is opening up other problems apart from diseases, students basically female (are responsible for 50% of food production in the world) are loosing 443 million school days fetching water from long distances and making it drinkable [Hwang ,2003, Crump, 2003]. No education or less education means an alleviation in economic status too, which in turn puts masses at risk of diseases due to non availability of money to afford safe water and better hygeine [Blakely et al, 2005, Clasen et al, 2006]. Potable water improvement and its management should involve collectors, users, researchers, policy makers and should be seen as a commodity with a reasonable cost [Gadgil, 1998]. Water is thus affecting economic status, education and food production [Savenije et al, 2009]. These aspects hence also becomes one of the subjects of study in this dissertation. The fact that, education is a must to better life and also to prevent water related diseases, is discussed in Chapter 6 of this dissertation with the help of a multivariate multiparameter regression model analysis on a survey in rural Nigeria, Africa.

There is a need to provide economic feasibility for small water supply system. This would point at efficient POU devices, with low material cost, sustainable, safe, easy to manufacture, negligible requirement of transport costs and low cost technology which is understandable to people and children with less education. To meet the Millenium Development Goals by the United Nations our removal technology should meet the criteria enumerated below [UN, 2006].

a. Is this technology of water filtration having a major impact on the social and personal lives of people?
b. Other than water filtration what other needs it will serve? For example, will it help to develop a business or will it help reduce diseases other than diarrhea?
c. Is this technology socially, politically and culturally encouraging ?
d. Does it really help in improving school hours for the women community and fill the gaps in knowledge ?
e. Does this help in income generation and job growth for communities and nations?

The developing coutries in Africa, Asia and South America are dotted with many recent projects for providing improved potable water after filtering most of the biological and chemical impurities. For example, ceramic water purifier has been introduced in

Cambodia, while in Nepal chlorination at home is adopted with Piyush chlorine solution, Kanchan filter has been used to fight arsenic in water and SODIS or solar water disinfection (developed in Switzerland) for small potable water quantities [Shakya et al, 2004, Shresta, R. R et al, 2004].

Ceramic filters have become a promising lowest cost option for needy developing countries to provide improved potable water (with 99% bacterial removal) at point of use [Brown et al, 2010]. The major feature is that these filters could be easily manufactured on site without much technical knowhow [Brown et al, 2010]. This increases accessibility of this technology for the common man living in rural areas of this world .

In recent studies of point of use household water treatment systems ceramic filter and bio sand filter were suggested as the most sustainable options for water purification in developing contries [Sobsey, et al, 2008].

The other major aspect is the question, what are the future pollutants and microbial contaminants of drinking water? Does evolutionary changes have any effects on these organisms to resist or defeat the chemical and biological techniques for removal and identification? It has been pointed out that use of epidemiological surveillance, its microbial causes and basic understanding of the microbial metagenome of the human body may help predict new microbial changes, adaptations and evolutions [Sobesy et al, 2009].

1.6 Objectives

Ceramic filter are very simple systems to understand, view and had less physical parts in comparison to bio sand filter. Here the purpose of this doing this research is in answering the following interconnected and interdependent questions

1. There has been a recent observation that flow rate of these ceramic filters increase with utilization time [Lantagne et al, 2010]. This may hint on a increase in pore size of these porous filters. Is the flow variation dependent on time of percolation through the filters. If this is true how the volume fraction of raw material

constituents influence this change in flow through this composite porous clay media.

2. This increase in pore size may be due to dissolution of chemical compounds on the surface of the clay ceramic porous filters [Halem, 2006]. This dissolution can be coupled with improvement in the pH of the filtrate. Is the dissolution affected by influence of temperature, chemistry of the influent fluid, chemistry of the ceramic vessels, flow changes with time and quantity of raw material constituents.
3. In the recent past, research has shown that the filters develop crack while in use as well as during transportation [Murcott et al., 2008]. This possibly provides us insight on breakage due to (pore size increase) enlargement of defects with time of filtration. How breakage is influenced by predictor variables like quantity of raw materials, microstructural geometry of the finally formed composite clay ceramic and strength characterestics at a specific temperature.
4. Finally it should be kept in mind that the common man at some location of the Earth is the end consumer of this filter. Hence continuing on this, an educative simplification of the manufacturing, characteristic and performance of the ceramic filter is dealt. This is done in order for the society of people with least literacy to understand the above mentioned complex processes and outcomes with ease. Cultural inheritance, economics and educational status is also to be considered to assess the feasibility of this filter in any specific society.

This would lead us to investigate interdisciplinary fields of material defects, flow and dissolution of porous media, strength characteristics, chemical thermodynamics and social sciences. So here in this dissertation clay ceramic frustum shaped filter is chosen to be studied and researched [Brown et al, 2010, Lantagne, 2001, Sobsey et al, 2008]. In order to study, we need to research on the materials and material science which would help us to provide sustainable solutions. The characterization of flow, material properties and structure of ceramic filters is thought to improve the effectiveness of ceramic filters for even the future emergent pathogens and pollutants.

1.7 Outline of this dissertation

To improve water quality, it is not only the design of the filter that matters but also the material used for manufacturing the porous media, their structural feasibility and sustainability, chemical composition of the geographically specific raw materials, location specific impurities in the water, people using the filter, their education levels and accessibility to better technology at reasonable cost may play a greater role [Salamanca-Buentello et al 2005].

A literature review on material, structure, flow variabilities and characteristics of the frustum shaped clay ceramic filters used around the world is provided in Chapter 2. Similar frustum shaped filters have been used in rural regions in developing nations around the world for providing improved potable water.

Chapter 3 provides an analysis of flow through the filters for point of use filtration of water. These filters have been manufactured from clay and organic material (sawdust). It also provides a simple, novel model to mathematically enumerate the complex physics of gravity driven water flow through these ceramic porous media filters. An analysis has been provided to predict the flow through the porous filter material with respect to independent variables of volume fraction of raw materials used for manufacture and time required for a volume of water to drain through a thickness of the ceramic porous media.

A structural analysis of the clay ceramic manufactured with variant fractions of constituents raw materials is described in Chapter 4. Prediction of fracture toughness of the clay ceramic as well as its stress bearing capacity is explained with multiple parameters of volume fraction of raw material constituents, geometry of the ceramic material specimen, applied loads on the notched clay ceramic specimen and also the orientation of the specimen with respect to its manufacture.

Chapter 5 deals with the leaching characteristics of filters. Data used in this study were collected by D. Van Halem in 2006 and documented in her MS thesis [Halem, 2006].

Three case studies are discussed in this chapter. First pH has been predicted as a function of time and material characteristics. Secondly pH increase had been predicted for shorter time spans using thermodynamic, electrokinetic and flow characterestics conforming to Onasager's prediction of interdependence of transport processes linked with the above mentioned parameters. Thirdly, an extension of the second case is studied for a time span of 5 weeks.

Chapter 6 examines the major impact of V shaped ceramic filters on the health as well as social and economic aspects of people at EwejeVillage , Odeda local Government Area, in Nigeria.

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Chapter 2: Literature Review on Clay Based Water Filters

Abstract:

Ceramic water filters are currently used around the world for point of use water filtration. This review characterizes the filters according to their shapes, material content, function and manufacture. A brief discussion on the effects of additive organic materials and their properties is provided. Effects of colloidal silver application on ceramic water filters and the mechanism of microbial inactivation are discussed. The literature on variations in flow through these ceramic filters with respect to different parameters is cited. Structural characteristics and breakage of the filter while in usage are also reviewed. The work of social groups and organizations for the dissemination and distribution of ceramic filters are briefly reviewed.

2.1 Ceramic Water filter: Positive Aspects and Reason for Feasibility

1. Clay ceramic business is well established around the world. Ample and cheap labor for clay work is available at local locations of manufacture [Brown et al, 2010].
2. Materials such as clay, diatomaceous earth wood chips/ sawdust, agricultural residues of rice, tea leaves/coffee grounds, corn/wheat flour, starch, charcoal, coal, powdered/activated carbon etc are readily available around the world at reasonable costs [Hwang, 2003, Klarman, 2009, Oyanedal-Craver et al, 2008, Nandi et al, 2007, Swanton, 2008].
3. Recent studies have shown an efficient removal of microorganisms from drinking water with the help of ceramic filters in general. It is also found that occurrence of diarrhea has greatly reduced in Africa, Asia and South America following use of ceramic filters for purifying potable water [du Preez et al, 2008, Sobsey, et al. 2007].

4. It is very important to note that social and cultural interaction and ties have played their part in the dissemination and distribution of ceramic filters. Social workers, nongovernmental organization and funding agencies played an important role in providing accessibility and contacts across international borders [Lantagne, 2001, du Preez, 2008, Brown et al, 2010].
5. From the review of more than 30 technologies for water filtration at point of use in developing countries, ceramic water filters are among the top 5 promising technologies [du Preez, et al, 2008, Sobsey, 2000]. Ceramic filters and biosand filters are the most sustainable technologies in the field of purification of drinking water in developing countries [Sobsey et al, 2008].

2.2 Classification of Ceramic Filters

a. Geometrical Shape parameters are cylindrical or candle, disk, pot or frustum or V shaped.

b. Base Material Parameters are clays for example, white kaolin, red terracotta, black clay, illite, montmorillonite and diatomaceous earth.

c. Additive Materials such as sawdust, wheat/corn flour, rice husk, coffee husk, tea leaves/coffee ground, starch(potato powder), grog, sand, iron oxide and colloidal silver (CS).

d. Functional characterization of clay ceramic filters

(1) Filtration of pollutant through screening and straining

(2) Removing odor and taste.

(3) Keeping the water cooler by energy transfer: The process of evaporation absorbs heat thus keeping the water inside the clay vessel cool.

(4) Storage of Water

e. Manufacturing Methods in local sites

(1) Potters Wheel (2) Manual or Mechanical or Servo-Hydraulic Press

MIT, Universities of Colorado, Tulane, West Virginia and North Carolina in the US, University of Delft, Stratcylde, etc. in Europe, USAID, UNICEF, Zamorano University in Honduras, Rafeal Landivar University in Guatemala, Institute of Hydraulic resources, Red Cross, Engineers Without Borders are some of the organization that have conducted research and proven the effectiveness of clay ceramic water filter [PFP, 2007, Franz, 2005, CN, 2007, Halem, 2006].

Some major ceramic water filters currently used around the world are summarized in Table 2.1.

Filters	Material	Additive	Coating	Cost	Microbial Efficiency (E.coli)	Flow Rate	Reference
Ceramic Filter , Australia	Clay	Tea leaves/Coffee grounds/ Rice husk	Nil	Nil	96.4-99.8	0.5l/h	Dr. Tony Flynn (ANU, Aust.)[Holtslag, 2010]
Siphon Filter , India	Clay candle (Pozzani or Stefani)	NA	Nil	$2.0	Depends on height of water	2-5l/h	[GWA, Holtslag, 2010]
Terafil disk Filter	Red Terracota Clay	Sawdust, Rice husk ash	Nil	$ 0.49	93-99.9%	1-11l/h	[Low, 2002]
Pelikan/ Indian Candle	white Kaolin clay	sawdust	Nil	$ 8.00-21.00	>3.9MPN/ 100ml	300-840ml/ hr/candle	Bajaj [Sagara,2000]
Nepal Candle	White Kaolin clay	Charcoal powder	Colloidal Silver (CS)	$4.07	99.9%	240-400ml/ hr	[Dies, 2003]

Continued Table 2.1

Continued Table 2.1

Capped Candle Filter, Nepal	Clay	Sawdust, Rice husk ash/Flour	C S	NA	99%	678ml/hr	[Dies, 2003]
Ceradyn	Clay	-	Nil	$190	98%	641 ml/hr	Katadyn®
Gravidyn	Clay	-	Nil	$160	98%	845 ml/hr	Katadyn®
Doulton	Diatomaceous Earth	Loose Carbon(Interior)	CS	$40	99.9%	1.34l/h	Franz, 2005
Disk Filter	Red Clay	Flour/Rice husk	CS	$2.00-3.00	99.9%	756 ml/hr	[Low, 2002]
Disk filter	Black Clay	Flour/ Rice Husk	CS	$ 2.00-3.00	99.3%	341ml/hr	[Low 2002]
Indu. For Poor Inc Filter	Sediment	Activated Carbon/Chlorine	Nil	$15	99.4%	9.5l/h	[Dies , 2003]
Kisii Filter Kenya/Burkina Faso/Niger	Kaolin Clay	Activated Coal	CS	$1.00	-	3l/day	[Traore et al, 2000, Fairwater.org, 2003]
Filtron/Kosim (frustum Shape)	Clay	sawdust	CS	$6.00	99-100%	1-2l/h	[Lantagne, 2001, Hwang, 2003, Swanton, 2008, Sobsey, 2010]
Filtron (Frustum Shaped)	Terracotta clay	sawdust	CS	$6.00	97.6%	1.7l/h	[Matellet, 2005]

Continued Table 2.1

Continued Table 2.1

RDI/CRC 2006	clay	Sawdust/ rice husk	CS	$7-10.00	97-99%	1-3l/h	[Sobsey et al, 2007, Brown, 2007]
Klarman tests(Filterpure)	clay	Coffee husk/Rice husk	CS	NA	96-97.69%	5-9l/h	[Klarman, 2009]
British Berkefeld(Zimbabwe/S. Africa	Kieselguhr Diatomaceous Earth	Ext. cover treated granular carbon	CS	$42-50	>99.99%	3.33l/h	Sterasyl™ [du Preez et al, 2008]

Table 2.1: Different filter systems: their filter element/media, effectiveness and cost.

Candle filters are prone to breakage and leakage from the joints [Clasen & Boisson 2006]. The filter parts are commonly produced away from places of usage (Bajaj filters from India, Ceramica Stefani filters from Brazil, Ceradyn and Gravidyn filters from Switzerland) and it can be difficult to obtain the replacement parts (Clasen et al. 2006). Transport of these brittle ceramics played a role in breakage of filters due to hairline cracks on the rim or body of the filters [Lantagne, 2001, Murcott et al, 2008]. In many ways, this characteristic could make the candle filter unsustainable.

Disk filters were plagued with leakage proving to be ineffective for longer use. A new disk variant was introduced by MIT in Nepal which stopped leakage and increased the microbial removal efficiency of disk filters [Matellet, 2005]. 65% of the 500 homes distributed with frustum shaped ceramic water filter, reported disuse due to filter breakage [Brown *et al.* 2007]. Since frustum shaped ceramic water filters can be manufactured locally, there is no need to access replacement parts in comparison to candle filters.

Recently pot shaped filter manufactured from coffee husks were found to break near their rim supports [Klarman, 2009].But these filters have not been having problems with maintenance or replacement.

2.3 Literature Review on Frustum Shaped Ceramic Filters

The InterAmerican Bank devised a methodology to find the best available clay filter for use in Nicaragua in 1981 [PFP, 2007]. Optimal flows, removal of micro organisms and cost were some of their criteria for choosing the best filter [Estrada, 2006].

Dr. Fernando Mazariegos of the Central American Research Institute manufactured the first pot-shaped ceramic water filter with a colloidal silver (CS) coating. He worked with Medical Assistance Program to train potters in Ecuador to make the filters, and soon other NGO groups followed [PFP, 2007].

Chlorine misuse in rural communities of Guatemala prompted the introduction of ceramic filters into homes. Ceramic filters rely on gravity to pass water through a porous medium [Estrada, 2006].

PFP is now an independent business with around 600 people, started by Mary Chapman in 1986. Its first technical liason officer Steve Earp hired Late Ron Riviera (ex-Sandanista employee) sociologist and a potter in 1989 to transform PFP for the international deprived [Estrada, 2006, PFP, 2007].

PFP thus provided a socially responsible assistance to potter groups and individual clay workers in their research for betterment in ceramic production thus preserving their cultural inheritance around the world [PFP, 2007].

PFP standardized the shape and size of Dr. Mazariegos filter in 1998 (Hurricane Mitch in1998) in the name "*Filtron*" and began mass production at Managua, Nicaragua for implementation in 1999 [Donachy, 2004]. Till 2006 PFP has factories established in Mexico, Bangladesh, Cambodia, Haiti, Guatemala, El Salvador, Srilanka, Nepal, Pakistan, Uzbekistan and Ghana reaching more than 2 million people [CM, 2007, Hagan et al, 2009].

In Africa mainly in Ghana, it is known as *Kosim* (in Dagbani dialect in (Oti-Volta)Gur Language " water served for the guests") [CM, 2007, Hagan et al, 2009, Swanton, 2008]. To enhance availability across borders and to reach the needy and economically backward communities without hassle, the filter has not been patented [PFP, 2007]. Pine sawdust [Klarman, 2009], rice husks [Hagan et al. 2009, Sobsey, 2006], flour [Oyanedel-Craver & Smith 2008, Klarman 2009], coffee husks [Klarman, 2009] are other locally available materials are used as a substitute to wood in different locations around the world. The other organic materials under test trails are potato powder, milled millet, peanut husks, and paper [Klarman, 2009].

2.4 General Manufacture Technique of Ceramic Water Filters

A very vague and crude process was performed in the rural locations where these filter were installed. A combination of clay and fine wood chips (porogen) is taken 1:1 ratio [Lantagne, 2001]. This mixture was then moistened with water in the form of 1-4 pound balls as shown in Fig 2.1 a and pressed into molds (greenwares) in a hydraulic press as enumerated in Fig 2.1 b[Cuff, 1996, Lee, 2001, Estrada, 2004].

Figure 2.1: Step by step manufacturing of ceramic filter from moistened balls of clay and sawdust blend.

Once press formed into frustum shapes they are retrieved out from the mold as shown in Fig 2.1 a and d. These are called greenwares. They are kept at room temperature to help greenwares lose water by drying them at room temperature as depicted in Fig 2.1 e. Once dried these greenwares are fired in a kiln to about 900°C. The sawdust is burnt off during the high temperature of firing, leaving behind the red colored clay ceramic filter with micro/nano pores in them [Lee, 2001, Hwang, 2003].

If the proportion of sawdust is changed in the mixture, the flow rate will also be affected because of the changes in geometry of the pores formed [Dies, 2003, Donachy, 2004]. The carbonate inherent in natural clay promotes the formation of pores of 1 micrometer in size. The reduced amounts of carbonates result in reduction of porosity [Cultrone et al, 2004]. Once fired, the filter is painted with colloidal silver (CS) in the inside and outside surfaces, thus preventing bio film formation on the filter [Sagara, 2000, Bielefeldt et al, 2005]. The CS solution has a silver concentration of around 110 ppm [Donachy, 2004]. The volume of CS applied on the ceramic filter is approximately 300mL [Donachy, 2004].

The clay ceramic filters potentially can be used for filtration for longer than 5 years with proper care and maintenance, although it is recommended to regularly replace the filter element every 1-2 years [Lantagne 2001; Campbell 2005].

2.5 Additive Characteristics and Their Variations and influences

2.5.1 Structural and Geometrical Feasibility of Frustum Shaped Ceramic Filters

There are geospatial variations on the filters manufactured at various locations around the world as shown in Table 2.2 below.

Filters	Top Diameter of Frustum in cm	Thickness of Wall in cm	Height in cm	Bottom Diameter in cm	Reference
Kosim, Ghana	31	1.35	24	1.6	[Matellet, 2006]
Bangladesh	56	3	23.7	48.4	[PFP, 2007]
Ghana	54	1.3	23.5	40	[Halem, 2006]
Cambodia	53	1.6	23.5	39	[Brown et al, 2007
Nicaragua	53	1.3	22	44	[Halem et al, 2007]
Dominica	52	1.4	23.4	42	[Klarman, 2009]

Table 2.2: Location specific variation in geometry for better efficiency of the PFP filters.

There has been a change in the micro structural properties as well as density is shown in Table 2.3 below,

Filter Material	Porosity in %	Density in gm/cm^3
Cambodia	43	1.37
Ghana	39	1.27
Nicaragua	34	1.3

Table 2.3: Location specific variation in micro structural geometry and density of the matrix manufactured from equal quantities of clay and sawdust [Source: Halem, 2007].

The quality and feasibility of the product always depends upon easy availability of components and manufacturing options. A presentation at the WHO conference in 2004 and in a report submitted to UNICEF by Sobsey and Brown, they presented some facts which were seen to be disadvantages faced by Potters of Peace filter [Sobsey et al, 2007]. Reduction in the clay to sawdust fraction and increase in the quantity of burnt organic material within the clay matrix decreased filter efficiency in filtering as well as structural feasibility [Lantagne et al, 2010].

Frequent ceramic water filter breakages with a frequency of about 2% per month after initial use were reported from Cambodia [Brown et al, 2006; Roberts, 2003]. Similarly Hwang in 2003 reported that 15% of the filters broke during a 6 month study revealing a flaw in filter design [Hwang, 2003]. In Dominican Republic , experiments with coffee husk as substitute to sawdust and rice husk , took a back stage as the filter made of clay and rice husk combination were not sturdy enough. It was found that they broke in 2 -3 weeks of water filtration [Klarman , 2009]. Major reason for breakage was non uniform blend of clay and coffee husk, even after a longer blending time [Klarman, 2009]. But filters made from rice husk in Cambodia by RDI were being used successfully by the people for more than a year [Sobsey, 2006, Hagan et al, 2009]. Uneven drying at the initial stages (greenware) of manufacture of the ceramic filter can help hairline cracks to develop which might be even difficult to see even after firing the greenwares [Cuff, 1996].

It is very important to know which type of organic material will suit filtration as well as for structural feasibility. This is only possible by trial and error techniques at the local level of production. This is due to chemical variability in clay mineralogy with geographical location, climate, time of formation and other local factors. Clays are formed from igneous, epigenic and hypogenic processes with time. Kaolinite is found in hot and humid location around the earth. For example, Ghana has kaolin type clay with much high iron content. General molecular formulae for kaolin found in South East Ghana was $Al_{1.8}\ Fe_{0.1}\ Mg_{0.1}\text{-}Si_2\ O_5\ (OH)_4 - Ca_{0.05}$[Sadahiro et al, 2006].

Clay soils in Managua in Nicaragua consist of organic diatomaceous volcanic clays with quartz, plagioclase, montmorillonite (smectite) and volcanic glass as major constituents [Swain, 1966]. It should be noted that organic matter is adsorbed on to clay and is a prominent part of clays. They have marked influence on properties of clay. General formula for montmorillonite (smectite) clays is $Al_{1.67}\ Mg_{0.33}\ Si_4O_{10}(OH)_2$. They are also found in different other chemical transformations namely Nontronite, Beidellite, Saponite and Hectorite [Worrall, 1986]. Chemical transformations do take place with change in temperature in clays [Worrall, 1986].

2.5.2 *Flow Characteristics*

The pore density distribution in the filter structural volume as well as porosity has a major effect on the flow characteristics and feasibility on the field [Hagan et al, 2009]. There were variations in flow between the filters in these global locations which is shown in Fig 2.2 below.

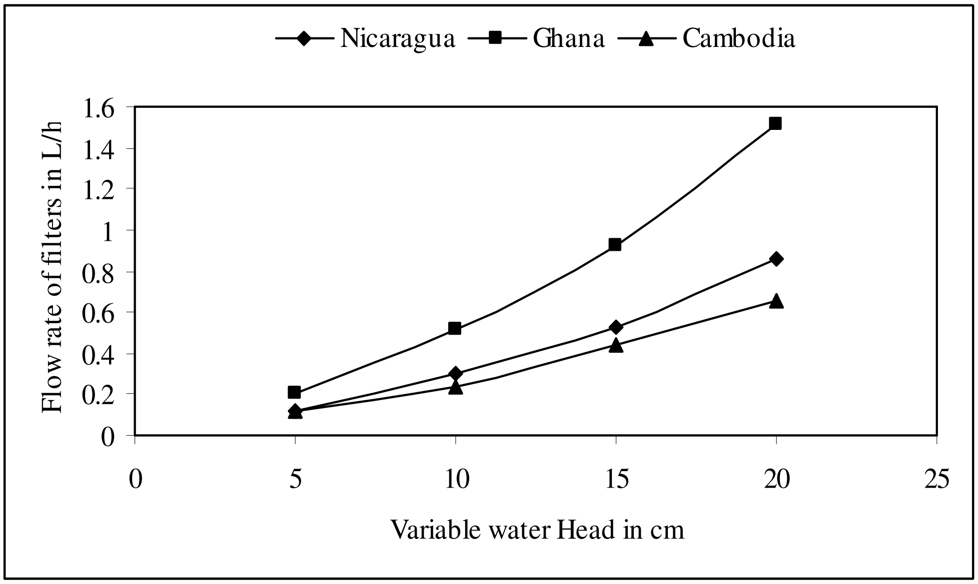

Figure 2.2: Flow rate as a function of water head in the filters [Bielefeld et al, 2005, Halem, 2006].

With increase in head of water, there is an increase in the flow rate through the porous ceramic filters. This is basically due to increase in porous surface area in contact with influent water. This is verified by the extension of the Darcy's Law for frustum shaped geometry [Halem, 2006].

According to Lee et al. (2001), there is an exponential relationship between hydraulic conductivity and the porosity of the ceramic with specific composition of clay and wood chips.

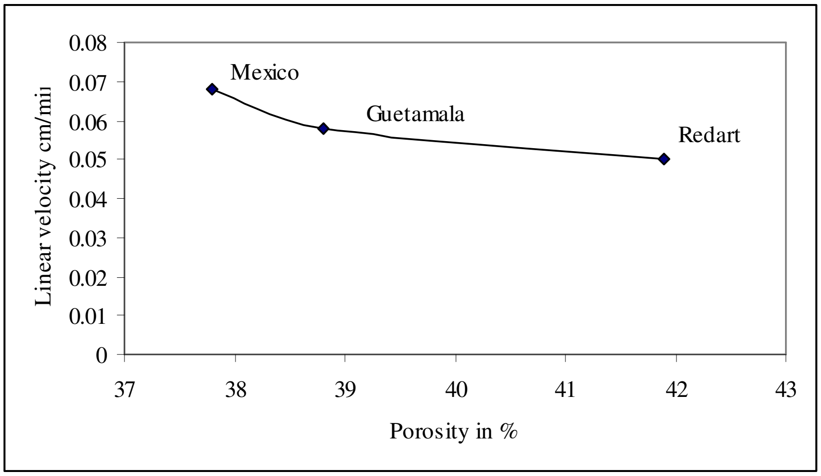

Figure 2.3: Particle velocity variation for clay ceramic water filters from Mexico, Guatamala and Redart [Oyanedel-Craver et al, 2008].

There is a decrease in velocity of flow with increase in porosity [Oyanedel-Craver et al, 2008]. There is an exponential increase in discharge as well as hydraulic conductivity in ceramic with an increase in the amount of organic carbon content [Lee, 2001]. The Potters for Peace filter is expected to have a flow rate between 1-2 l/h (Lantagne 2001). Potters for Peace Filters with flow rates more than 2 l/h can remove 100% of total and fecal coliforms (Lantagne 2001).

Experiments in the Dominican Republic reported 50% increase in flow rate after approximately 120 hours from the first use of the filter [Klarman, 2009]. This increase may be due to agglomeration of additives to influence pore clustering, which would result in detachment of clay matrix and organic additives of rice husks or coffee husks [Klarman, 2009].

Disuse of filters was reported from some quarters in Cambodia since ceramic filters were having very low filtration rate [Brown et al, 2007]. Similar dissatisfaction was reported from Bolivia where around 30% of ceramic filter users commented on low flow rate [Clasen et al, 2006].

The other parameter affecting filters were recontaminations. Recontaminations are caused by the stagnancy persisting inside the filter and long usage. This recontamination could be one of the major factors which may reduce the efficiency of the filter [Brown, et al, 2007]. This is the reason why microbial removal was having a diminishing character with time, until unless cleaned to remove the microbial deposition within the filter after a specific time of utilization.

2.5.3 Microbial Effectiveness of the Porous Clay Ceramic Filters

Studies by Duke *et al.*(2006), Lantagne (2001), Mattelet (2005), Sagara (2003), Roberts (2003), Van Halem (2006), and others have suggested that ceramic water filters provide microbiologically improved water to users as indicated by the log removal values reported in these studies. The porous clay ceramic filter realized greater than a 4-log reduction of protozoa (Eg. Giardia lamblia and Cryptosporidium parvum) but was not effective for viruses such as MS2 Coliphages [Lantagne, 2001]. The PFP filters in practice proved to be 95 % efficient instead of a 99.99% field performance results in terms of E. coli removal [Sobsey, 2006]. Napotnik and others in 2009 at Leigh University confirmed that microbial removal efficiency of both Potter for Peace filters and Filterpure filters (Dominican Republic) were almost similar.

A study spanning from 1993 to 2005 found that the ceramic filters could reduce diarrhea cases by almost half the rate. In 2001, Lantagne found that the Filtron ceramic water filters were 100 % efficient in removing bacteria. There was a decreased removal of coliphages which were of 0.02 micron size. In Guatemala, ceramic filter use led to 53% reduction in diarrheal incidence [Brown, 2007].

2.5.4 Colloidal Silver Application

Oyanedel-Carver and Smith proved that rather than method of application the amount of colloidal silver (CS) applied was more effective in bacterial removal. In 1993, Heinig studied silver deposited on alumina ceramics. The suggestion was that silver-alumina surfaces promoted catalytic interaction with oxygen, which resulted in bactericidal activity. He found that bacteria and viruses were decimated on contact without any release of metals into the water [Jain et al, 2005]. Bactericidal properties are affected by the availability of the ionic silver to be in contact with the bacteria in the clay-water system [Kumar et al, 2004, Magana et al, 2008]. This was confirmed by the X-ray photoelectronic spectroscopy (XPS) done on the clay samples exposed to E.coli infested water. Electrochemical deposition of silver over carbon or charcoal helped better deactivation of microorganisms [Kumar et al, 2004]. For example, silver in atomic stage absorbs oxygen and act as a catalyst to bring about oxidation. Nascent oxygen absorbed onto the surface of silver ions in solution reacts with the sulfhydryl (-S-H) groups surrounding the surface of bacteria and viruses to remove the hydrogen atoms, causing the sulfur atoms to form an –Organic -S-S –Organic bond [Shashikala et al, 2007]. This blocks respiration and causing the microbe to expire [Kumar et al, 2004]. This electrochemical surface deposition is shown in Eq. 1 below [Shashikala et al, 2007].

$$Organic - S - H + O + H - S - Organic \xrightarrow{Ag-Car.Cov.Al_2O_3} Organic - S - S - Organic + H_2O \tag{1}$$

Three mechanisms of bacterial deactivation of silver are catalytic oxidation, reaction with cell membranes and binding with the DNA of organisms to prevent unwinding respectively [Shashikala et al, 2007].

Silver from the colloidal silver binds to the cell membrane of bacteria, increasing size and cytoplasm contents, and cell membrane. These result in cell lysis and death, as the silver substitutes compounds in the cell membrane that are required for cell membrane stability [Donachy, 2004, Hungbao et al, 2007, Lantagne, 2001]. It should be noted that CS leaches through the filter thus decreasing surface concentration of CS with time of filtration [Donachy, 2004, Halem, 2006].

2.5.5 Organic Materials and Contaminants

Organic materials are available in almost every location on earth and would be affordable for anybody to use. Treated and untreated organic material has been used by earlier researcher in removing pathogen, metals, dyes and other chemical such as sulfides, nitrates etc from water successfully [Bulut et al, 2007, Huang et al, 1985, JETRO, 2006, Memon, et al, 2007, Shukla A, et al, 2002, Shukla S et al, 2005]. Organic materials referred here are sawdusts, for example, walnut sawdust, pine sawdust, deodar sawdust, agricultural food processing residues such as rice husk, coffee husk and biomass found in water such as Chlorella vulgaris, Clodophara crispata, Zoogloea ramigera, Rhizopus arrhizus and Saccharomyces cerevisiae [Ajmal et al. 1998, Bulut et al. 2007, Nourbakhsh. et al, 1994, Shukla et al. 2002, Shukla et al. 2005].

Lignin is a natural component of clay present in a non-separable way with aromatic components and structure with ether-oxygen linkages [Himmel, et al 2007, Worrall, 1986]. The chemical groups within cellulose are, the hydroxyl groups (OH^-) in radial orientation and the aliphatic hydrogen atoms in axial positions [Himmel et al, 2007]. Organoclays can be useful sorbents for organic contaminants and heavy metals in water [Oyanedel-Craver, et al, 2006]. Zero valent iron bind with viruses helping virus removal from aqueous solutions [Bloem et al, 2009, You et al. 2005].

On heating plant biomaterials such as wood, rice husk etc, they form negatively charged carbon surfaces [Busscher et al, 2008]**.** Lignin side chains oxidize to form $COOH^-$ groups forming humic acid. Lignin and humic acid are highly colloidal materials [Himmel, et al 2007, Worrall, 1986].

Carbons formed after firing remove bacteria such as E.coli from water through attractive Lifshitz–Van der Waals forces despite electrostatic repulsion between negatively charged bacterial cells and clay-carbon surfaces. Activated carbons had been earlier used for removing cobalt ions from water [Huang et al, 1985].

Chromiun ions have been removed from aqueous solution using clay sawdust composite [Shukla et al, 2002]. Organic materials (treated wood/ treated peanut hull act as activated carbon) has been used earlier to remove lead and mercury in ionic state from aqueous solutions [Namasivayam, et al, 1993, Raji et al, 1996]. Heat treated sawdust of Mangifera indica (Mango tree) was used to remove Copper ions from water [Ajmal et al, 1998].

Adsorption of materials such as metallic ions and heavy metals on to organic materials are governed by ion exchange and hydrogen bonding [Shukla et al, 2002]. Metal ion M^+removal through adsorption is shown below in Eq. 2 and Eq. 3[Shukla et al, 2002].

Ionic Exchange takes place as shown [Shukla et al, 2002]

$$2(R - COH) + M^{2+} = (R - CO)_2 + 2H^+ \tag{2}$$

Hydrogen Bonding may take place as enumerated [Shukla et al, 2002]

$$2R - COH + M(OH)_2 = (R - COH)_2M(OH)_2 \tag{3}$$

Here R is the component of organic material contribution [Shukla et al, 2002]. The pH of the water is an important parameter in the adsorption process in case of clay ceramic surfaces which are made of basic alumino -silicates and organic materials [Ajmal et al, 1998, Elliot and Huang, 1981]. Sorption of cadmium, lead and zinc in aqueous solutions decreases with increase in pH [Oyanedel-Craver et al, 2006]. With thermal affects there are many changes in organic materials such as plant cell structures and material composition. It is to be noted that even sound waves specific to a location may affect the plant cells by changing their chemistry and structure [Kashap, 2005].

In early 19th century a single Kalash or Kolshi filter was used in parts of Nepal and India with Thoria based clay, *Loganiceae* plant paste, and charcoal [Jayaraman, 1993, Hwang, 2003, Dies, 2004, Murcott et al, 2002, Shresta et al, 2003]. The paste is used for water clearing, purification and filtration aid for several centuries in India, Nepal, Africa, Sri Lanka and Burma. The seeds of this *Loganiceae* plant in India are used to purify water for drinking [Mallikharjuna et al, 2007, Gupta et al, 1992]. The polysaccharides and proteins of the bark and stem have bio-flocculant properties [Adinolfi et al, 1996, Jahn, 1994] for removing cadmium, mercury and other toxic heavy metals and which may bind to gold, silver, cobalt, copper and nickel. It is also proved that the seeds are useful to clean long-lived isotopes from nuclear wastes [Jayaraman, 1993].

Moringa oliefera or Moringa seed extracts can achieve 98% removal of fecal coliforms under optimal conditions [Jahn, 1986, Jahn, 1988]. It is commercially available as a completely biodegradable Moringa coagulant, under the commercial name PhytoflocTM [Jahn, 2009]. Hence it should be noted that organic material used in ceramic filters play an important role in purification of water and there are numerous options to modify and use organic materials with specificity related to geo-spatial contaminants.

2.6 Organizational and One to One Knowledge Transfer

Social marketing plays a major role in helping this project to initiate, evolve and progress. The frequency of use of filters by individuals and households cannot be controlled because of many unpredictable conditions such as job nature, behavior, education and untimely social events. It is found from a survey in Bolivia that from the total filters distributed, 70% were only regularly used [Clasen et al, 2006]. It is always important to study the setting of the society to which the filter is going to be distributed. This will avoid non uniform filter performance against microbes as well as will help better incorporate the protective effects of the filter to the society [Clasen et al, 2005].

In recommendation to UNICEF, educational and behavioral change programs were proposed for implementation to disseminate knowledge on contamination due to improper use [Brown et al, 2006]. This also supports the household intervention of diseases [Clasen et al, 2007].

Ceramic water filters due to their low cost and better performance can be better options for providing safe water for disaster relief operations [Clasen et al, 2006]. Time for the study or survey is a major parameter which helps quantify health response, but may be cut short due to prohibitive costs and participant fatigue [Brown et al, 2008]. This is also influenced by climatic condition prevailing at such study locations. Other characters such as breaking community ethics, losing trust of the survey community may also hinder studies [Brown et al, 2008].

2.7 Conclusion

A brief review of clay based filters is discussed. Multitude of materials used in the manufacture of these filters has been discussed. Filters have been characterized by size, shape, function, manufacturing methods and material type. The chemistry of clay and sawdust has been briefly reviewed.

Dissolution or wasting away clay filters during filtration improved alkalinity of the filtrate. The effect of pH on adsorption has been cited. Adsorption mechanism of different organic materials has been reviewed. Flow rate was found to vary with time of utilization. Cracking or breakage of clay filters had been seen at various locations. They occur while transportation, manufacture as well as usage.

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Chapter 3: Theoretical and Experimental Investigation of Water Flow through Porous Ceramic Clay Composite Water Filter

Abstract

Water flow through point-of-use porous ceramic water treatment filters have been theoretically analyzed in this chapter. Filters tested were manufactured by combining low cost materials namely, clay and sawdust. Three filters with distinct volume fractions of clay to sawdust (75:25, 65:35 and 50:50) were tested. Sintered clay filters casted in frustum shapes were structurally characterized using mercury intrusion porosimetry. A linear increase in porosity with volume fraction of sawdust was observed.

Flow experiments were carried out at constant room temperature and pressure. Potable tap water was used in these studies. Flows through filters occurring with drop in the head of water under gravity were statistically analyzed. Discharges through the filters were predicted with respect to independent variables of time for cumulative discharge and volume fraction of sawdust used for manufacturing the filters. The experimental data analysis predicts a multiplicative influence of time and volume fraction of sawdust respectively, on discharge from the filters. The results demonstrate a new theoretical approach for prediction of flow in similar types of heterogeneous porous media as discussed in this chapter.

3.1 Introduction

Water is a basic requirement to sustain life for human beings and animals around the world. There is a scarcity of potable water in developing countries. The people in these countries are not able to afford costly water purification technologies and devices at point-of-use (basically in their households) due to low per capita earnings [Islam (1996)]. Hence, there is a pressing need for low cost, locally produced, highly reliable and efficient water purification technologies and devices.

The requirements for filtration of water vary from region to region based on impurity characterizations such as, microbes [Wegmann, Michen and Graule, 2008], chemicals [Boujelben,Bouzid, Elouear, Feki, Jamoussi, and Montiel, 2008; Genc-Fuhrman, Mikkelson and Ledin, 2007], metals [Lin, Wu and Lai 2008; Hatfield, Annable, Cjo, Rao and Klammler, 2004; Ahmedna, Marshall, Hussieny, Rao and Goktepe, 2004], silt [Genc-Fuhrman, Mikkelson and Ledin, 2007] and organic matter [Lin, Wu and Lai, 2008]. Some of the basic point-of-use methods and devices for removing the above mentioned impurities are chlorination, solar disinfection, ceramic filters and bio sand filters (specific in removal of metallic impurities) [Sobsey, Stauber, Casanova, Brown and Elliott, 2008].

Ceramic filters made of clay augmented with state of art technologies such as nanofiltration, can be used for providing cheap potable water in the developing countries [Hillie, Munasinghe, Hlope and Deraniyagala, 2003]. Past research and social work has helped millions of people across the globe to take advantage of clay composite ceramic filters for water purification [Sobsey, Stauber, Casanova, Brown and Elliott, 2008; Hillie, Munasinghe, Hlope and Deraniyagala, 2003].

Similar type of clay ceramic water filters were encouraged and distributed for usage in developing countries by a nongovernmental organization named Potters for Peace [PFP 2006, Donachy, 2004]. It was a low pressure water filter relying on gravity for its operation. It was manufactured by combining locally available clay and sawdust (hence, a clay composite) and had a frustum shape. This filter has not been patented [PFP (2006)] and has been providing pure potable water in developing countries across Central America [Donachy, 2004, Lee et al. 2001; Halem 2006], West Africa [Halem 2006; Swanton 2008] and South Asia [Sobsey, Stauber, Casanova, Brown and Elliott, 2008; Swanton, 2008; IRIN, 2008; Brown and Sobsey, 2006]. A 99-100 % microbial removal was registered during several field tests [Franz, 2005; Lantagne, 2001]. Health reports through 1993-2003 from Central America showed a 50% reduction in waterborne diseases after usage of these filters [Donachy, 2004; Brown and Sobsey, 2006].

A general methodology for manufacturing this clay composite was followed and disseminated by Potters for Peace [PFP, 2006, Donachy, 2004]. The composite filters were manufactured from moistened suspensions containing clay and sawdust in a 50:50 ratio by volume [Lee et al., 2001; Halem 2006]. Due to the plasticity of moistened clay composite, it could mold under stress to any shape as required. The filters were casted in the shape of a frustum or flower pot [Donachy, 2004]. Sintering these filter molds to around 900^{o}C introduces numerous pores in the mold serving its filtration capabilities [Lee et al., 2001; Halem, 2006; Franz, 2005; Hwang, 2003; Dies, 2003; Oyanedel-Craver and Smith, 2008].

Extensive literature is available on several empirical developments to analyze flow through these porous ceramic clay composite filters [Lee et al., 2001; Halem, 2006; Lantagne, 2001; Hwang, 2003].

Being a porous device it is supposed to follow Darcy's law which is given by [Dagan, 1989],

$$Q = -\kappa A \Delta h / L \qquad (1)$$

where Q is the discharge from the porous media, A is the surface area which holds the water, Δh is the change in head of water due to gravity flow, K is the hydraulic conductivity of the porous structure and L is the thickness of the porous media through which the water needs to percolate [Dagan, 1989].

Hydraulic conductivity K is defined as [Dagan, 1989],

$$\kappa = \gamma k / \mu \qquad (2)$$

where μ is the viscosity of water at a given temperature, γ is the specific gravity of water at that particular temperature and k is the intrinsic permeability defining the porosity and interconnectivity of the porous media [Dagan, (1989)]. The flow velocity V through the medium can be written in the form

$$V = \frac{-k\left(\sum_{i=1}^{3}\frac{\delta p}{\delta x_i} - \gamma\sum_{i=1}^{3}\frac{\delta h}{\delta x_i}\right)}{\mu} \tag{3}$$

where $\sum_{i=1}^{3}\frac{\delta}{\delta x_i}$ is the gradient function and p is the pressure and h is the variable head of water above the porous media [Chen, Huan and Ma, 2006]. Numerical models as well as image analysis has been used to study permeability [Arab, Semma, Pateyron, and El Ganaoui, 2009, Chattopdhyay, Knight, Kapadia and Sarkar, 1994, Chen, Huan and Ma, 2006].

Halem in 2006 found that, there is a change in flow rate with change in location of the manufacture of the filter. Experiments were carried out on filters from Cambodia, Ghana and Nicaragua and were made with clay to sawdust ratio of 50:50 [Halem, 2006]. From these observations it was envisaged that filters made with locally available clay and sawdust at these locations were influenced by their respective material properties. These affected micro-structural properties such as porosity, for example filters from Cambodia, Ghana and Nicaragua have 43%, 39% and 34% porosity respectively [Halem, 2006]. The corresponding mean discharges from filters in the above mentioned countries were 0.76 l/hr, 2.41 l/hr and 0.85 l/hr respectively [Halem, 2006]. Similar empirical tests were conducted by researchers on samples from Mexico, Guatemala and Redart and concluded randomness in the behavior of similar volume fraction filters [Oyanedel-Craver and Smith, 2008].

In another study, it was concluded that porosity varies with increase in volume ratio of plant material [Lee et al., 2001]. Porosity was predicted to be a linear polynomial fit with 99.45% accuracy as

$$y = 43.05 - 0.4865x + 0.0127x^2 \tag{4}$$

where y is the porosity of the filter sample and x is the volume ratio of plant material [Lee et al., 2001]. A study analyzed influence of sintering temperature on porosity variations in clay ceramic filter containing Kaolin clays [Nandi, Uppaluri and Purkait, 2008]. This study predicted a decrease in porosity with increase in sintering temperature in the range 850-1000^{o}C [Nandi, Uppaluri and Purkait, 2008]. There would be a large influence on the properties of the final sintered product due to the shape, compositional materials used, processing environment and thickness [Bazant, 1999, Chattopdhyay, Knight, Kapadia and Sarkar, 1994, Worrall, 1986].

From the above discussion it is clear that a range of parameters influence the flow behavior through heterogeneous porous formations. Hence there is a requirement to identify the major parameters influencing discharge through non uniform porous media as discussed above. Earlier researchers have tried to use analytical models for the description of porous flows such as random walk models with Markovian chain based theories [Borgne, Dentz, and Carrera, 2008].

The present study identifies and discusses the major predictor variables that can influence the flow through clay composite ceramic filters. A statistical multi-parameter model elaborates the novel approach discussed later in this document, which would help us in understanding the material and transient effects on the discharge from frustum shaped filters and any other filter with a known geometrical structure.

3.2 Materials and Processing

3.2.1 Raw Materials

The composite filters were manufactured by combining specific volume ratio of materials namely, sawdust and clay. Sawdust was obtained from a local saw mill (Hamilton Supplies, Hamilton, NJ), containing 80% oak and 20% cedar as constituents. It was sieved manually using a 35-1000 micron mesh.

Clay supplied by Resco Products Inc. Pittsburgh, PA. USA, is a combinational mix of illite and kaolinite clays.

3.2.2 Manufacturing Process

Volume fraction of clay and sawdust, in accordance with our requirements were mixed together in a commercial blender (Model A-200, The Hobart Manufacturing Company, Troy, OH). The mixture was blended for about 10-15minutes to accomplish uniform mixing. The blended composite is mixed with water in small volumes under continuous mixing. Clay to sawdust mixes of 75:25, 65:35 and 50:50, ratio by volume, requires about ~ 1.8-2 litres of water to be made into a 12 lb (5.443 kg) dough ball, enough to manufacture a filter. These ratios were chosen to study extremes in cumulative percolation time (basically from 5-10 hours to a week in similar filter variants).

Axial press forming is used to provide the dough a shape. This can now be called the greenware in language of a potter [Cuff, 1996]. In this study, the green ware produced was frustum shaped. A 50 ton (45,359.237 kg) hydraulic press (TRD 55002, Torin Jacks Inc, Ontario Canada) was used for this purpose.

The press used a metallic frustum mold to form the greenware of requisite dimensions. The inner dimensions of the frustum were: length of axis, 26cm, lower base diameters, 20cm and upper base diameters, 23cm. The frustum wall and base have a thickness of 0.5cm and 1cm respectively.

For each of the volume fraction of sawdust discussed in this document, 6 filter greenwares with uniform geometric dimensions were manufactured. The greenwares were kept at room temperature for drying. Time taken for drying the greenware varied (5-8days) for different constituent volume ratios.

After drying the greenwares were sintered in a gas and electric kiln (Ceramic Arts Department, Princeton University, Princeton, NJ). The filters were then pre-heated to 450-550^{o}C for three hours in a gas kiln to burn off the saw dust [Cuff, 1996, Maritan, Nodari, Mazzoli, Milano and Russo, 2006]. After slow cooling to room-temperature, the filters were re-heated to 955^{o}C in an electric furnace. The initial heating rate of 50^{o}C per hour was increased to about 100^{o}C per hour beyond a furnace temperature of 200^{o}C. The filters were sintered for 5 hours at the peak temperature of 955^{o} C. They were then furnace cooled in air to room temperature.

Once the firing is completed the filters are left in the furnace for 5-10 hours to cool down. Greenwares were transformed into structurally rigid red colored composite ceramic filters, ready for use in experiments.

3.3 Experimental procedure

3.3.1 Material Analysis

The material formed after sintering the greenware should be structurally distinct owing to the variations in its basic constituents. Mercury intrusion porosimetry is used to measure the porosity of the ceramic composite structure of the filter.

Small chunks of material samples with a dimension of approximately ~3mm x 3mm x 3mm each were cut from each of the ceramic composite filters used here. Each of these three compositionally different samples was separately poured into the experimental chamber (the penetrometer) of the Micrometrics Autopore III: 9400 digital analyzer used to calculate material porosity. Generally, the chunks occupied about one-half of the penetrometer bulb volume. The analyzer follows a two step pressure analysis test on the ceramic porous sample, taking advantage of the non-wetting behavior of Mercury.

The main aim of the experiment is to find the effect of the volume ratio of the basic material constituents on porosity of the sintered clay ceramic material. Micrometric Autopore III: 9400 digital analyzer plots a pore size distribution curve and calculates the average porosity of the material.

3.3.2 Flow Experiments

Many researchers have done filtration tests focusing the long term effects on the flow behavior [Donachy, 2004; Halem, 2006; Swanton, 2008; Brown and Sobsey, 2006; Lantagne, 2001]. It is necessary to understand the flow behavior at microscopic time scale to establish variability due to material modifications and surface interactions taking place during the transient flow through filter media.

The sintered filters of each volume fraction were fully saturated with water by dipping them in a water bath containing purified water (Barnstead/Thermolyne, EASYpure uv/uf, Model D8611) for about 12 hours. This is done to simulate the actual discharge from the ceramic composite filter in a fully working condition.

The experimental setup as shown in Fig. 3.1 consists of three major components. First, a ceramic composite filter filled with water. Secondly, a vessel for collection of discharged filtrate from the filter.

Figure 3.1: Model experimental setup for flow experiments conducted for different filter variants.

Experimental setup was covered by plastic wrap to prevent evaporation as well as external influences and impurities. Finally, the filtrate collection vessel sits on a load cell (Model LSC 7000-50, Omega Engineering Incorporated, Stamford, CT).The load cell was connected to a LabView card (NI PCI-6259, National Instruments, Austin, TX) via a 68-Pin Digital and Trigger I/O Terminal Block (CB-68LP, National Instruments, Austin, TX) to a LabView software (Version 8.0, National Instruments, Austin, TX).

The data acquisition (DAQ) system can be used to calculate the amount of filtrate water collected at a resolution of one millisecond. The data are collected in a format to assist with the study of the flow analysis and is recorded as follows: The filtrate volume accumulated in the measuring vessel is recorded every second for the first 20 minutes of the start of filtration. Secondly the readings are accessed at every minute for the next 3 hours. After this, data are collected at every 3 minutes until the last drop of water within the filter.

The complete draining of water under gravity from within a fully filled composite ceramic filter is termed as one experiment. These experiments are conducted consecutively three times, one after the other, on each of the 6 filters manufactured for the different clay and sawdust configurations (75:25, 65:35 and 50:50 by volume).

A C- S (Volume of Clay in % – Volume of Sawdust in %) and C-S-E (Volume of Clay in % – Volume of Sawdust in % -Experiment Number) notation has been used in this document to describe the filters and their corresponding experiments.

It is very important to note that time has been a factor in constant and variable pressure filtration models [Johnston, 1995]. Similarly it has also been found that permeability has been a factor in the models for percolation through porous media [Hatfield, Annable, Cjo, Rao and Klammler, 2004; Dagan, 1989; Johnston, 1995].

The objective is to establish the basic variables and their mutual interactions influencing the flow rate of the filtrate from the ceramic composite filters. Thus the experiment focuses on the material constituents and their qualitative interactive effects on the flow through the heterogeneous system used in this study.

3.4 Results

3.4.1 Porosity Variations

The porous structures for ceramic composite filter materials with three compositions (75:25, 65:35 and 50:50 ratios by volume) of clay and sawdust were analyzed. The pore sizes in all the samples showed a wide range distribution within 0.001µm to 100µm as plotted in Fig. 3.2, 3.3, and 3.4.

The density of number of pores with sizes within 0.001µm to 1µm was observed to be very high in all the samples tested irrespective of its constituent quantities.

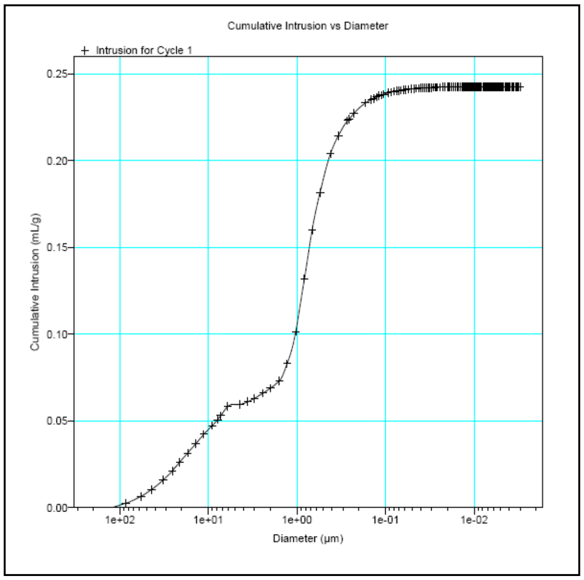

Figure 3.2: Pore size distribution for the sample from the circular base of the 75-25 frustum filter

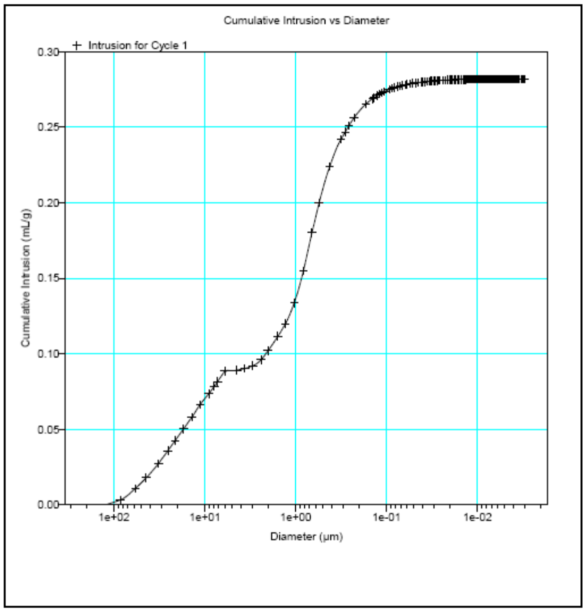

Figure 3.3: Pore size distribution for the sample from the circular base of the 65-35 frustum filter.

These random pore size distributions may introduce non uniform variation in porous media flow behavior. This variability throughout the filter would suggest development of probabilistic models for predicting flow through the ceramic composite filter.

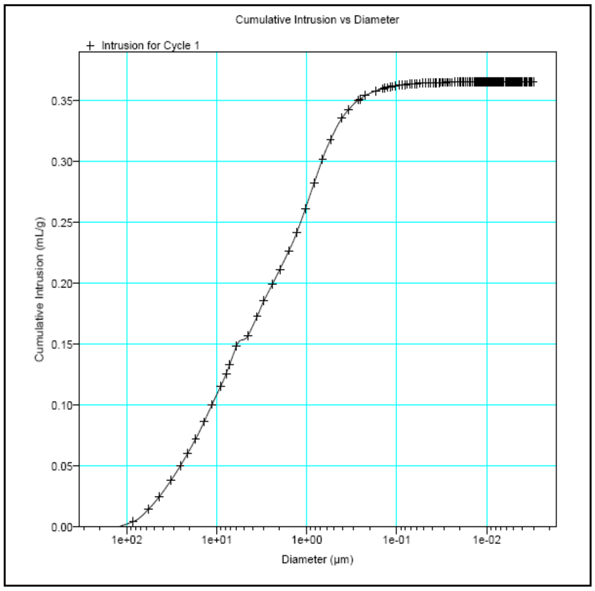

Figure 3.4: Pore size distribution for the sample from the circular base of the 50-50 frustum filter.

The variations in average porosity were observed with the changes in the volume ratio of the constituents for the manufacture of the filter. These average porosity values in percentage were an output calculated by the Autopore III: 9400 digital analyzer.

The plot in Fig. 3.5 implies that a straight line equation may be used to fit the increase in average porosity with increase in sawdust content by volume used for manufacture of these three filters. This also supports the explanation of influence of carbonate content on increasing porosity [Cultrone G., Sebastian, Elert, Torre, Cazalla and Rodriguez-Navarro, 2004].

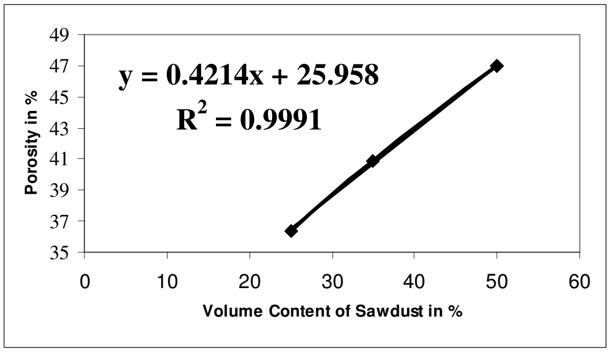

Figure 3.5: Linear equation fit for an increase in porosity with increase in sawdust for filters with 25%, 35% and 50% of sawdust by volume.

Porosity variations with location as observed in Fig. 3.6 may be due natural modifications in the raw material type and its configuration with respect to the geographical environment prevailing at that location.

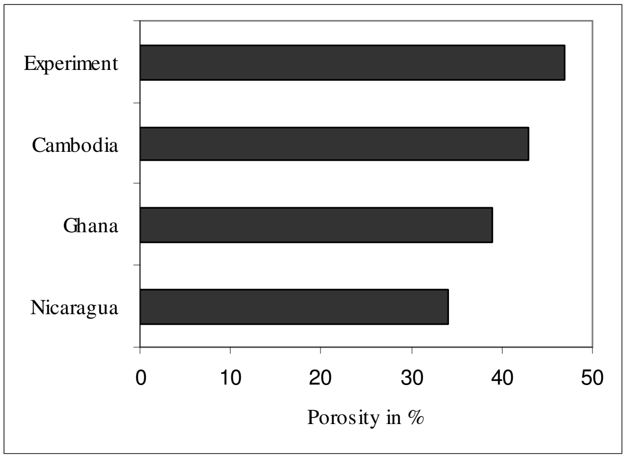

Figure 3.6: Comparative study on porosity variations (of the circular base of the 50-50 frustum filter at different locations and experiments conducted in this study) [Lee et al., 2001; Halem, 2006; Lantagne, 2001].

3.4.2 Statistical Modeling of Discharge through Ceramic Filters

A series of filtration experiments simulating water percolation through ceramic composite filters were done. These filters distinct in material composition had varied structural porosities as shown in Fig. 3.5. The initial filtrate volume measurement from all the three distinct filters 50-50, 65-35 and 75-25 has been plotted in the Fig. 3.7.

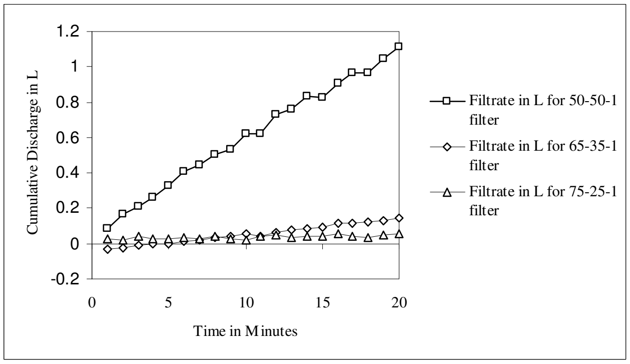

Figure 3.7: Behavior of cumulative discharge from the filters at the initiation of water percolation.

3.4.3 Theoretical Development

From Fig. 3.7, the discharge or filtrate volume can be seen to follow a rising trend with time. Nonlinearity is also detected by visual inspection of the rising curve. The filtrate volume or cumulative discharge Y_i is a random function of time X_i for *i= 0, 1, 2…n.* The total filtrate volume Y_n , accumulated and collected, is a result of a finite discharge from a filter in a specific interval of time. The two successive discharges vary each other by a random ratio or a transfer function. This can be written as

$$\frac{Y_i}{Y_{i-1}} = X_i^{b_i} \quad \text{, for } i = 1, 2 \ldots \text{n} \qquad (5)$$

Eq. 5 is a stochastic model, showing the relationship between the cumulative discharge Y_i at step *i*, from the previous cumulative discharge at step *i-1* and transfer function X_i^{bi}. This transfer function X_i^{bi} contains the effect of the predictor variable X_i at step i of the stochastic model [Soboyejo, 1965]. Since there are n predictor variables in the generalized multi-parameter stochastic model; Eq. 5 represents only one of the steps for $i = 1, 2 \ldots n$. The generalization has been elaborated in Appendix A. It can be inferred from Fig. 3.7 that filtrate volume Y_i at each and every time step is incremented independently.

There are wide deviations in the discharge during the three consecutive experiments on each of the filters discussed here. This is shown in Fig. 3.8, Fig. 3.10 and Fig. 3.12 for filters manufactured with 50-50, 65-35 and 75-25 clay to sawdust by volume respectively.

To remove the existing nonlinearity, it is required to make linearizing transformations [Soboyejo, 1965; Benjamin and Cornell, 1970]. This transformation provides us with a new response variable.

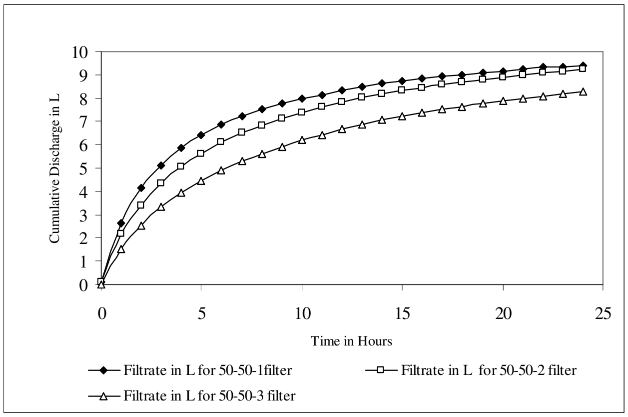

Figure 3.8: Discharge from three consecutive experimental runs on a 50-50 filter.

It is known that the curves drawn in Fig. 3.8, Fig. 3.10 and Fig. 3.12 may be characterized using discrete time stochastic birth process [Soboyejo, 1965; Soboyejo, 2006]. This was confirmed by performing regression data analysis on the basis of principles in experimental statistics [Soboyejo, 1965; Benjamin and Cornell, 1970; Ang and Tang, 2007; Cox and Miller, 1967].

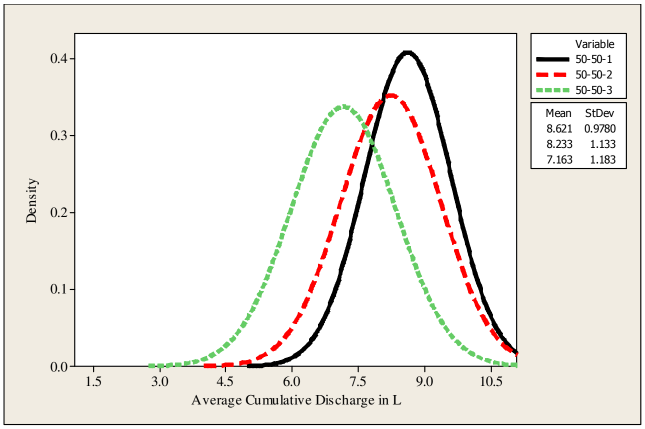

Figure 3.9: Histograms and normal distribution of cumulative discharge for consecutive experimental runs on the 50-50 filter.

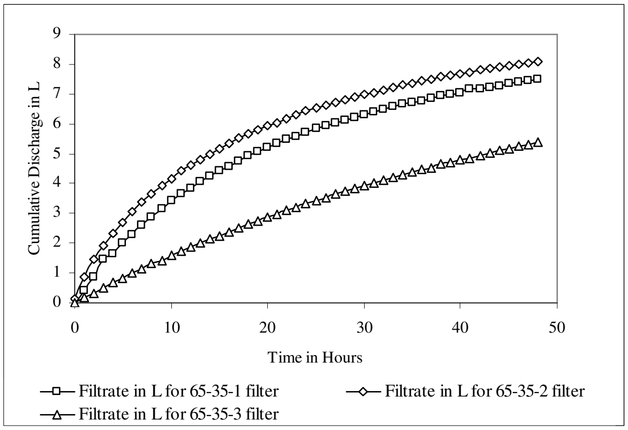

Figure 3.10: Cumulative discharge from three consecutive experimental runs on a 65-35 filter.

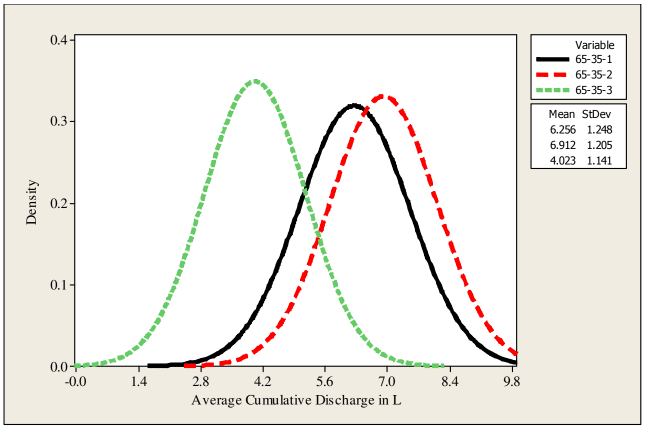

Figure 3.11: Histograms and normal distribution of discharge for consecutive experimental runs on the 65-35 filter.

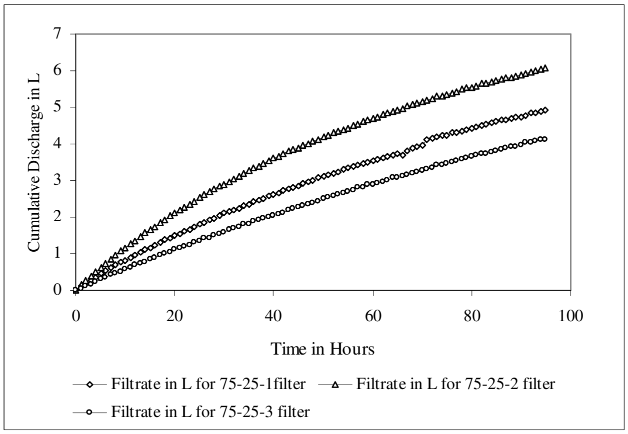

Figure 3.12: Discharge from three consecutive experimental runs on a 75-25 filter.

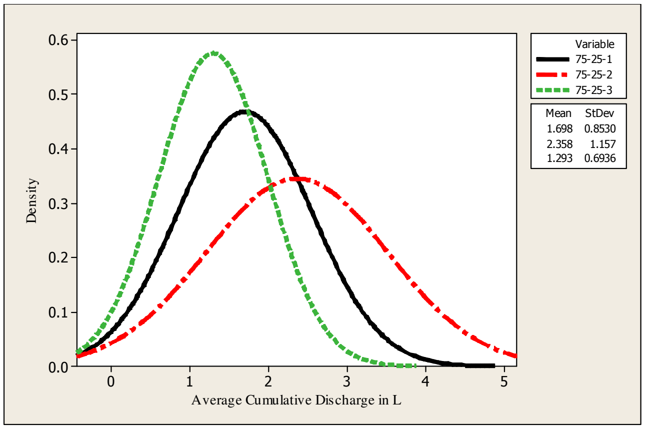

Figure 3.13: Histograms and normal distribution of discharge for consecutive experimental runs on the 75-25 filter.

If the variables are transformed in the following manner,

$$G_i = \frac{X_i}{Y_i}$$

The random variable G_i jointly characterizes the random variable X_i and Y_i. The relationship between the transformed random variable and X_i, is expressed as a straight line as given below [Soboyejo, 1965, Soboyejo, 1973].

$$Y_i = \frac{X_i}{a_i + b_i X_i} . \qquad (6)$$

where X_i for $i = 0, 1, 2, \ldots n$ and for $i = 1$ denotes time t. Here a_i and b_i are model parameters and are found to characterize individualistic material behavior and time respectively and is elaborated in the Appendix A [Soboyejo, 1965, Soboyejo, Ozkan, Papritan and Soboyejo, 2001]. The transformation can be used model the behavior of the flow through the ceramic composite filter with better values for the coefficient of determination, R^2 [Soboyejo, 2006]. This would help in determining a better empirical relationship for the flow through each of the individual filters.

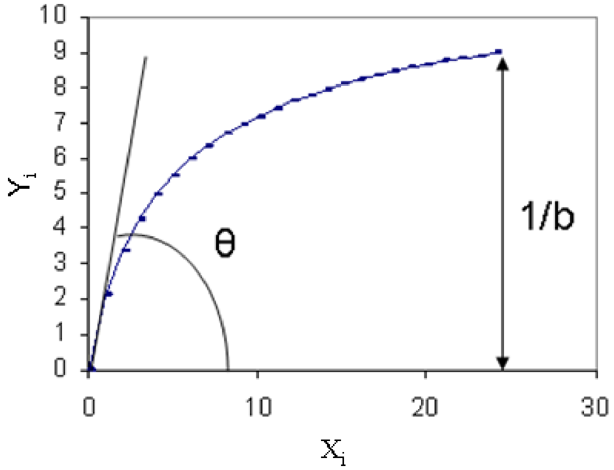

Figure 3.14: Description of the specific birth process model proposed in Eq. 6 for *i=1* [Soboyejo, 2006].

Rate of change of Y_i can be written as the first derivative of the Eq. 6 as [Soboyejo, 2006],

$$\frac{dY_i}{dX_i} = \frac{a_i}{(a_i + b_i X_i)^2} \tag{7}$$

From Fig. 12, at $X_i = 0$, for i= 1, we have

$\frac{dY_i}{dX_i} = \frac{1}{a} = \tan\theta$ where the derivative is also given as the arc tangent of the angle θ formed by each of the rising curves plotted in Fig. 3.8, Fig. 3.10 and Fig. 3.12 respectively and as elaborated in Fig. 3.12. Eq. 6 may again be expressed as

$$Y_i = \frac{1}{\frac{a_i}{X_i} + b_i} \tag{8}$$

As X_i tends to infinity, the equation 8 reduces to the form [Soboyejo, 2006],

$$Y_i = \frac{1}{b_i}. \tag{9}$$

This value of discharge may define the extrapolated value of the asymptote of each of the curves in Fig. 3.8, Fig. 3.10 and Fig. 3.12 respectively at requisite time durations [Soboyejo, 2006].

3.4.4 Flow Variation in Each Filter within Discrete Experiments

From Fig. 3.9, Fig. 3.11 and Fig. 3.13, it is seen that that flow behavior of 50-50 filters is different in comparison to 65-35 and 75-25 filters. The mean value of the filtrate volume decreases with number of experiments for all the 6 50-50 filters tested. This is shown from Fig. 3.9 as

$\overline{Y}_{50-50-1} > \overline{Y}_{50-50-2} > \overline{Y}_{50-50-3}$.

In this regard, the mean discharge values for 65-35 and 75-25 filters follow a different behavior as plotted in Fig. 3.11 and Fig. 3.13 and is explained below as,

$\overline{Y}_{65-35-1} < \overline{Y}_{65-35-2} > \overline{Y}_{65-35-3}$ and $\overline{Y}_{75-25-1} < \overline{Y}_{75-25-2} > \overline{Y}_{75-25-3}$.

Apart from these variabilities, the discharges from each of the experiments were characterized by discrete time stochastic processes. The discharge from the experiments done on 50-50 filters can be expressed as

$$Y = \frac{X_1}{0.432 + 0.0943X_1} \tag{10}$$

where the model parameters a and b are 0.432 and 0.0943 respectively. The statistical coefficient of determination for the model was found to be R^2 =99.9% from statistical regression analysis performed in Minitab Statistical Software (Version 15, Minitab inc, State College, PA).

Similarly a birth process model also predicts the discharge through the 65-35 filters with coefficient of determination of 99.7 % as

$$Y = \frac{X_1}{2.25 + 0.098X_1} \tag{11}$$

where the model parameters *a* and *b* are 2.25 and 0.0943 respectively. The discharge from the 75-25 filter specimens has been found to follow a birth process with a coefficient of determination of 99% as shown below

$$Y = \frac{X_1}{10.9 + 0.0855X_1} \tag{12}$$

where the model parameters a and b are 10.9 and 0.0885 respectively.

3.4.5 Discharge Variability due Multiple Parameters

From Fig. 3.7 and Fig. 3.15, it is very clear that each of the filters behave separately. The average discharge for three filters with different manufacturing material compositions has been plotted in Fig. 3.15. All the curves follow a rising trend with decreasing slopes with increase in time irrespective of their compositional variability.

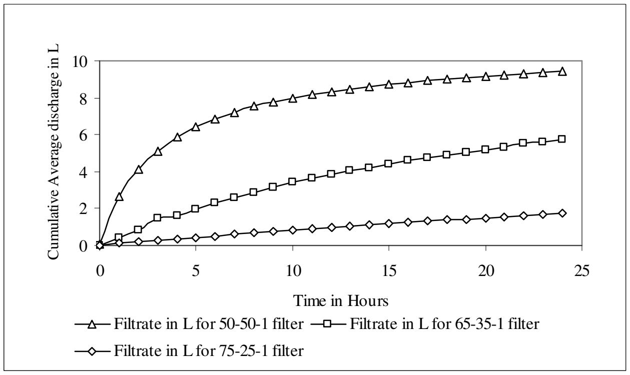

Figure 3.15: Measurement of discharge from three distinct filters for a duration of 24 hours.

The Eq.6 is extended to a multi-parameter model to predict filter discharge on the basis of time and compositional variability. Here the stochastic process occurring within the filter system is represented in Fig. 3.16.

From Fig. 3.16, the multi-parameter model may be written as

$$G_i = a\prod_{i=1}^{k} X_i^{b_i} \tag{13}$$

where the model constants of *a, b_i* for *i= 1.2……k*, are derived by regression analysis on the experimental data. It should be noted that derived transformation of multiplicative variables are also multiplicative variables [Benjamin and Cornell, 1970].

The model indicates the lognormal influences of naturally occurring materials such as sawdust and water on the process [Limpert, Stahel and Markus, 2001]. The inherent non linearity due to the influence of the natural variables is removed by taking natural logarithm on both sides of Eq. 13. This can be expressed as

$$\ln G_i = \ln a + \sum_{i=1}^{k} b_i \ln X_i \tag{14}$$

The Eq. 14 would be used for finding the model constants a_i and b_i. Multi-parameter regression analysis is carried out with two major random variables to predict the percolation though the ceramic composite filter.

Mean value of G_i is expressed as [Ang and Tang, 1975, Benjamin and Cornell, 1970]

$$E(G) = a\prod_{i=1}^{k} E(X_i^{b_i}) \tag{15}$$

The second central moment of the lognormal distribution representing G_i may be expressed as [Ang and Tang, 1975, Benjamin and Cornell, 1970]

$$Var(G) = \sum_{i=1}^{k} b_i^2 \left[\frac{\sigma_{X_i}}{X_i}\right]^2 G_i^2 \tag{16}$$

It is found that there is no correlation between the random variables considered in this multi-parameter model hence the covariance term Cov (X_i , X_j) tends to zero. Also, the coefficient of determination, R^2, provides the combined influence of the independent parameters in this model [Haldhar and Mahadevan, 2000].

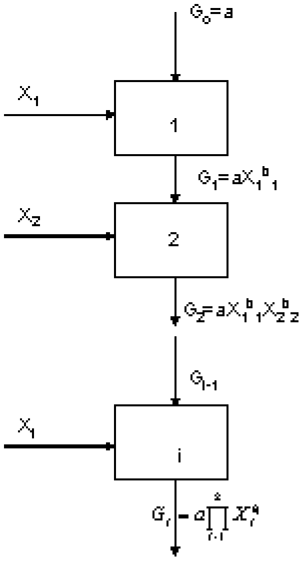

Figure 3.16: Diagrammatic representation of a stochastic process influenced by multiple parameters [Soboyejo, 2006].

In this model $G_i = X_1 / Y_i$ and the predictor variables are X_1 (time) and X_2 (Volume ratio of sawdust).

The variation of G_i was examined and modeled to a linear lognormal equation in Eq. 13 with a square of the multiple-correlation coefficient, R^2 of 95.1%.

$$G = 11.4 \times X_1^{0.32} X_2^{-3.02} \qquad (17)$$

Eq. 17 may, therefore, be used to characterize the percolation through the filter manufactured with a known volume ratio of material constituent, at a specific time.

3.5 Discussion

Average cumulative discharge Y_i for the three different filters discussed here, is seen to increase with increases in amount of saw dust, X_2 as shown in Fig. 15. The stochastic birth process behavior shown by the discharge from these porous clay ceramic filters is theoretically elaborated in Appendix A1.

A vast difference in percolation behavior within consecutive experimental trials was noted for 50-50 filters with respect to other filters discussed in this study. This distinctiveness may be attributed to the individual influences of clay and sawdust content on the structure of the filter material with time X_1 for cumulative discharge process in each experimental trial.

The variability for cumulative discharge in each filter of a particular type (50-50, 65-35 or 75-25) with respect to X_1 is well established and fairly determined.

It is known that there is negligible statistical correlation between the two variables X_1 and X_2 considered in this problem as discussed in the results above. In view of this, the effect of variability between these predictor variables is extremely low and insignificant.

The variabilities in this problem can be attributed to variability in material constituents and also variability arising from the manufacturing or production processes.

From Eq. 17, it can be seen that coefficient b_1 of the predictor variable $X_2$is much larger when compared to coefficient b_2 of predictor variable X_2. This clarifies the higher influence of predictor variable X_1 than X_2 on flow behavior in the clay ceramic porous media filter system with a specific volume fraction of sawdust [Bulmer, (1979), Soboyejo, (1965)].

Variability due to material constituents are much higher than possible variability arising from the effect of time. Therefore the variance or error of prediction of cumulative discharge with respect to time is much smaller than the error due to volume fraction of material constituents in this study. An analysis of variance is carried out to elaborate this notion. From Eq. 16 and Eq. 17, it can be further stated that,

$$Var\ G \approx (b_1)^2 Var(X_1) + (b_2)^2 Var(X_2) + (b_3)^2 Var(X_3) \qquad (18)$$

Much elaborate discussion on Eq. 18 is given elsewhere and will therefore not be given in this article [Soboyejo, 1968, Soboyejo, Ozkan, Papritan and Soboyejo, 2001].

Var (G) is the error in the prediction of the new transformed variable and Var (X_1) and Var (X_2) are variance or error expected in individual predictor variables X_1 and X_2 respectively. From empirical data analysis and calculations we have

$$Var\ G \approx (b_1)^2 Var(X_1) + (b_2)^2 Var(X_2)$$

$$0.848 \approx (0.32)^2 0.6703 + (-3.02)^2 0.0812 \qquad (19)$$

It is important to note from Eq. 19 that compositional material variability ($(b_2)^2 Var(X_2)$) is much higher than variability arising from the influence of time X_1 for cumulative discharge ($(b_1)^2 Var(X_1)$).

This in turn confirms that error of prediction of cumulative discharge with time is much smaller than the error due to the amount of manufacturing material constituents of the filter dealt in this study. Hence, investigations need to be performed to assess the variability in average cumulative discharge between individual clay ceramic filter variants and also within each individual variant filter.

From Fig. 3.9, Fig. 3.11 and Fig. 3.13, it is found that each of the three types of filters tested here have cumulative discharges following the central limit theorem and have approximately equal standard deviations respectively. These are the basic assumptions of an ANOVA test [Walpole and Myers, 1993].

The basic hypothesis was that mean average cumulative discharge for all the filter variants in this problem are equal to each other. This investigation was carried out with one way analysis of variance with a 95% confidence interval using Minitab software (Version 15, Minitab Inc., State College, PA). The results are as depicted in Table.3.1.

<table>
<tr><th colspan="6">Average Cumulative Discharge vs. Volume Fraction of Sawdust (VF) in the porous clay ceramic filter</th></tr>
<tr><td>Sources of Variation</td><td>Sum of Squares</td><td>DOF</td><td>Mean Squares</td><td>F-Statistic</td><td>p value</td></tr>
<tr><td>Between VF</td><td>449.10</td><td>2</td><td>224.55</td><td>118.16</td><td>0.00</td></tr>
<tr><td>Within VF</td><td>131.12</td><td>69</td><td>1.90</td><td></td><td></td></tr>
<tr><td>Total</td><td>580.22</td><td>71</td><td></td><td></td><td></td></tr>
</table>

Table 3.1: ANOVA Test.

From Table 3.1, a very large value of F-statistic indicates that there is a large difference in mean average cumulative discharge values between the three filter variants studied in this chapter and not much variations within a particular filter variant.

This rejects our hypothesis and confirms with the discussion on Eq. 19. It is also seen that p value is lower than 0.05, proving that volume fraction of sawdust is a very significant contributor to the efficient percolation behavior of the filter.

The results of the ANOVA and Eq. 19 are supported by the multi-parameter regression analysis done as is enumerated in the Table 3.2 below.

Predictor Variables\Model Coefficients	$\bar{a}$	$\bar{b}_1$	$\bar{b}_2$	S	R^2
X_1	-0.627	0.68	0	0.889	0.287
X_2	-11.4	0.68	3.02	0.206	0.962

Table 3.2: Multiplicative model for porous ceramic clay composite water filter discharge (L) using variables X_1- time and X_2- Volume Fraction of sawdust.

Here in Table. 3.2 denote the values of the coefficients $\bar{a}$ and $\bar{b_i}$ of the predictor variables X_1 and X_2. From Fig 3.17, the potential process capability of the model for X1/Y is found to be at par with the industry bench mark for CPL=CPU=Cpk =1.33.

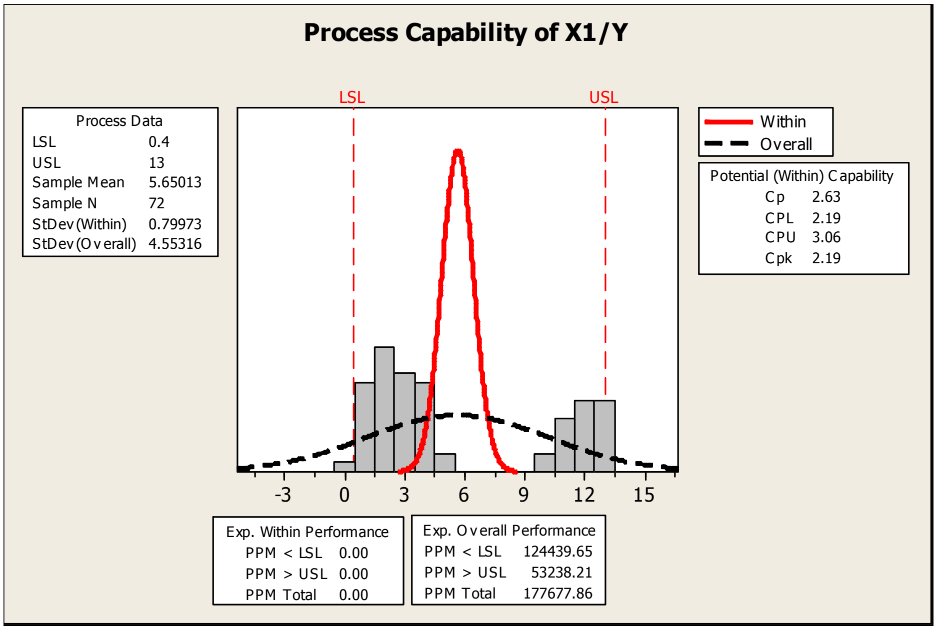

Fig 3.17: Sensitivity Analysis for the Eq. 17.

Here CPL measures the closeness of the mean value of X1/Y to its lowest possible value, CPU measure the closeness of the mean value of X1/Y to its highest possible value and

Cpk denotes the lower value between CPL and CPU. The PPM (parts per million) total for expected “within" performance is 0. This means that approximately 0 out of a million will not meet the specification limits. This analysis tells you that your model is fairly capable. Thus from Fig 3.17, it is found that flow process through the clay ceramic filter can be better predicted with this model in Table 3.2. It is found that, in comparison to the predictor parameter of time, the volume fraction of sawdust used in the manufacture of the porous ceramic clay composite water filter influences the discharge *Y* the most. This is true for within a family of filters with different compositional mixes of clay and sawdust by volume.

The results of the present investigations are applicable only to the material configurations, size and shape tested. Whenever the need arises to have any change in material configurations, shape and size the new technological principles developed in this present study, can be applied in order to obtain the new model for this new configuration, shape and size. It is intended to extend the present technology to include new configurations, shapes and sizes in future investigation.

3.6 Conclusion

1. The micro-structural properties are highly influenced by the quantity of the material used in the manufacture of the ceramic composite filter. A linear trend in the variation of porosity is seen with change in volume ratio of the material constituent used to manufacture the filter.
2. It is very important to note that a single parameter stochastic birth process model for estimating the flow through a porous clay ceramic vessel with a specific material composition has been developed. Discharge follows a specific behavior depending on the amount of material constituent used in the manufacture of that filter.
3. A lognormal multi-parameter model has been proposed for predicting the flow behavior through a heterogeneous system with varying volume ratio of materials.

4. The contribution of time, volume ratio of the manufacturing materials as well as type of process play an important role in efficiently defining hydrodynamic behavior of a non uniform porous media.

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Chapter 4: Experimental and Multivariate Stochastic Analysis of Mechanical Strength of Controllable Porous Clay Composite Ceramic Matrix

Abstract

A series of frustum shaped ceramic wares were produced by uniform mixing, axial press forming and sintering of clay and sieved sawdust combined at various volumetric compositions. The strength tests were conducted on specimens taken from the circular base of the frustum shaped ceramic filter ware. The specimens and their orientations were characterized on the basis of plastic flow of wet clay - sawdust suspension observed during the press forming operation. The addition of organic fibers (sawdust) resulted in porosity augmentation, density reduction and nonlinear decrease in fracture toughness, flexural strength and compressive strength in the ceramic ware. Stochastic lognormal regression and variance analysis is used to estimate uncertainties in predictor variables related to volumetric constituents, micro-structural porosity and fracture loads. Assessments on the relative contribution of each predictor variable are enumerated here. The models developed in this chapter can be used for the characterization of strength of similar clay ceramics at specific temperatures.

4.1 Introduction

Clay contributes towards the operational and textural properties of several materials [Henry, 1955]. Clay is an important constituent in glasses, porcelain or china ware, pottery, refractory, cement and engineering ceramics [Davidge, 1979]. The addition of water, organic and inorganic (acid and bases) chemicals influence the rheology, plasticity and forming characteristics of clays [Norton, 1942]). With addition of water, clays thicken after a time, and may be sheared back to flow. These behaviors of thixotropy affect the degree of plasticity of clays [Kremenchutskaya et al., 1981]. Degree of plasticity varies for different clay mineralogy and may be defined by flow curves given by Bingham flow equation shown below:

$$\sigma_s - \sigma_v = A\gamma + B\gamma^2 \qquad (1)$$

Where: $\sigma_s\ and\ \sigma_v$ are the shear stress and yield value of clay-water system and γ is the shear rate of the clay-water system (Worrall, 1986). Here A and B are:

$$A = (g + \eta)b/(a\gamma + b) \text{ and } B = a\eta/(a\gamma + b) \qquad (2)$$

Where: η is the absolute viscosity of the clay–water system, and a and b are coefficients of the above model. Here g is the constant related to degree of flocculation. Degree of plasticity is independent of water content but may be influenced by additives to the system; for example, sand addition is found to decrease plasticity. Similar influences may occur with addition of organic and inorganic materials. From the Biblical days, vegetable material (straw) has been added to lumps of mud to positively influence its mechanical properties [Sines, 1969]. Organic extracts (from hay) have been used as an additive for improving strength (Acheson, 1904). Clay with or without additives are transformed to ceramics after sintering, having variant chemistry, texture, material characteristics and use. For example, kaolinitic clays can be chemically transformed to meta-koalin at 450°C, while at 925°C to silica-aluminum spinel and at 980°C to mullite [Chakraborty, 2003, Papargyri, 2006, Worrall, 1986].

Clay ceramic materials are stronger in compression compared to tension [Barsoum, 2003, Mazdiyasni, 1990, Green, 1998]. Ceramics are ionic or covalent bonded materials with large atomic lattice spacings making them brittle in nature. Fracture stress in ceramics (glass) is inversely proportional to the dimension of the fracture surface. This may be represented as fracture stress $\sigma = M * r^{-1/2}$, where r is the dimension of the mirror, mist and hackle crack fracture surface and M is a constant for this surface with the units of stress intensity [Johnson and Holloway 1966, Soboyejo, 2003]. The constant M is also proportional to K_{Ic} critical stress intensity for Mode I crack propagation in poly crystalline ceramics [Kirchner, 1979, Mecholsky, 1976]. An extension to the above equation defining fracture stress (Johnson and Holloway 1966) was developed by Kirchner and Kirchner (1979) through an integration of non-uniform stress fields and crack geometry.

Kaolinitic clay have been reinforced with inorganic fibers to make a new class of ceramics [Papargyri, 2003], the mechanical properties of which depended on the fiber and matrix structure and corresponding volume. These additives may be in the form of particulates, whiskers or fibers [Barsoum, 2003]. Organic materials, such as sawdust from pine, oak, deodar, flour, tea/coffee ground and husks, groundnut shells and paper, have been used as additives in clay-based ceramics used for water filtration [Klarman, 2009]. Organic starch when mixed with alumina in certain ceramics led to reduced strength and Young's modulus, but an increase in porosity [Dorey, 2002]. Improvement in the volume of organic material decreased the structural feasibility of ceramic porous water filters [Lantagne, 2010]. Hence it appears that the volume of reinforced materials as well as natural organic matter influence strength and micro-structural properties of ceramics.

Clay ceramics are porous hence they are two-phase materials [Davidge, 1979, Henry, 1955]. The pores exist throughout the micro-structure and are nucleated during the manufacturing and sintering processes [Nandi, 2008, Oyanedel-Craver, 2008]. The pore morphology variations account for over 20% of the variation in the actual strength of ceramic substances [Askeland and Phule, 2003]. Strength was found to decrease with an increase in volume of the porous composite material [Danzer, 2008, Dorey, 2002]. This was due to pore clustering or the increase in the occurrence of pores in a larger volume than in a smaller volume [Danzer, 2008]. These pores may also be categorized under forms of defects.

Strength characteristics relating to the weakest defect, defect density and the size of the brittle material as a whole, have been expressed using Weibull's model [Evans, 1978, Batdorf, 1978]. Thus it is noted that porosity plays an important role in defining the strength of the ceramic materials. Weibull models do not take into account the correlations existing between the predictor variables [Bazant, 1999, Soboyejo, 2001]. Random scatter of strength in brittle ceramics cannot be described by a single Weibull modulus [Cannillo, 2000, Fok, 2000]. Complete representation of the material property behavior was supposed to be enumerated by a three parameter Weibull, but not for porous clay ceramics [Fok, 2000, Papargyris, 1998].

The Modulus of elasticity and strength of alumina–starch composite ceramic was modeled using the Spriggs Equation [Dorey, 2002]. The micro-structural properties change with change in process parameter and time [Nandi, 2008, Peterlik, 2001]. These influence the final strength characteristics of ceramics [Nandi, 2008, Peterlik, 2001]. Fracture toughness was predicted using a linear relationship given by adding the product of porosity of the material with unit cell fracture toughness, and applied stress, respectively [Cherepanov, 1979]. Stochastic models reflect the realistic nature of monotonic load application upon a defined area and its relationship with notch size [Cheng, 1992]. Thus from the literature above, it can be noted that mechanical properties are developed by a step-by-step process with time.

It is also seen that parameters such as volume of raw materials, porosity as well as type of materials (organic or inorganic), temperature, water content while manufacturing and plasticity influence the strength of brittle ceramics. A stochastic multivariate multi-predictor approach has therefore been proposed to predict the compressive as well as toughness characteristics of clay composite ceramics. Based on the probabilistic strength models developed here, the errors or variances have been examined and quantified. Correlation existing between the predictor variables influencing strength has been studied.

4.2 Materials and Processing

4.2.1 Raw Materials

Clay ceramics were manufactured by combining specific amounts of natural materials, namely sawdust and clay. Sawdust was obtained from a local sawmill and contained 80% Oak and 20% Spanish cedar [Plappally, 2009]. The sawdust dimensions were controlled by manual sieving, using a 35-1000 mesh (Hamilton Supplies, Hamilton, NJ). The clay referred to here is a mix of Illite and Kaolinitic clays, commercially available from Cedar Heights Redart Airfloated Clay, Pittsburgh, PA, USA.

4.2.2 Manufacturing Process

Clay and sawdust were mixed in volume ratios of 75:25, 65:35 and 50:50. Uniform mixing was done with a commercial blender (Model A-200, The Hobart Manufacturing Company, Troy, OH). The blended composite consumed approximately 2 liters of water for molding a 6-kg (12-lb) dough ball, required to manufacture one porous clay ceramicware (PCCW) filter. The blended composite was pressed formed to a frustum shape using a hydraulic press (TRD 55002, Torin Jacks Inc, Ontario, Canada). The formed composite at this stage of manufacturing is refered as greenware [Cuff, 1996]. Clay particle size and shape, mineral composition, organic matter content, soluble salts and ionic elements influence the molding properties [Norton, 1942].

Each of the greenwares had an axial length of 26 cm and base diameters of 20 cm and 23 cm, respectively. They also had a thickness of 0.5 cm and 1 cm on the frustum wall and base, respectively. Fig.4.1 shows a greenware taken out of the mold pattern.

Figure 4.1: Greenware wrapped in plastic being removed from the mold pattern.

Fig. 4.2 shows a two-dimensional representation of the step-by-step axial press forming procedure. The plasticity of the clay-sawdust water suspension plays a major role in forming the circular base as well as the walls of the frustum-shaped greenware. The arrows in Fig. 4.2 show the direction of transport of the wet, clay-sawdust suspension during forming. This plastic flow may align the sieved, sawdust fibers along the direction of flow [Papargyri, 2006]. Quick changes occur in the micro-structures (chemical transformation, pore formation or densification) during liquid phase sintering due to rapid material transport through the fluid [Kang, 2005].

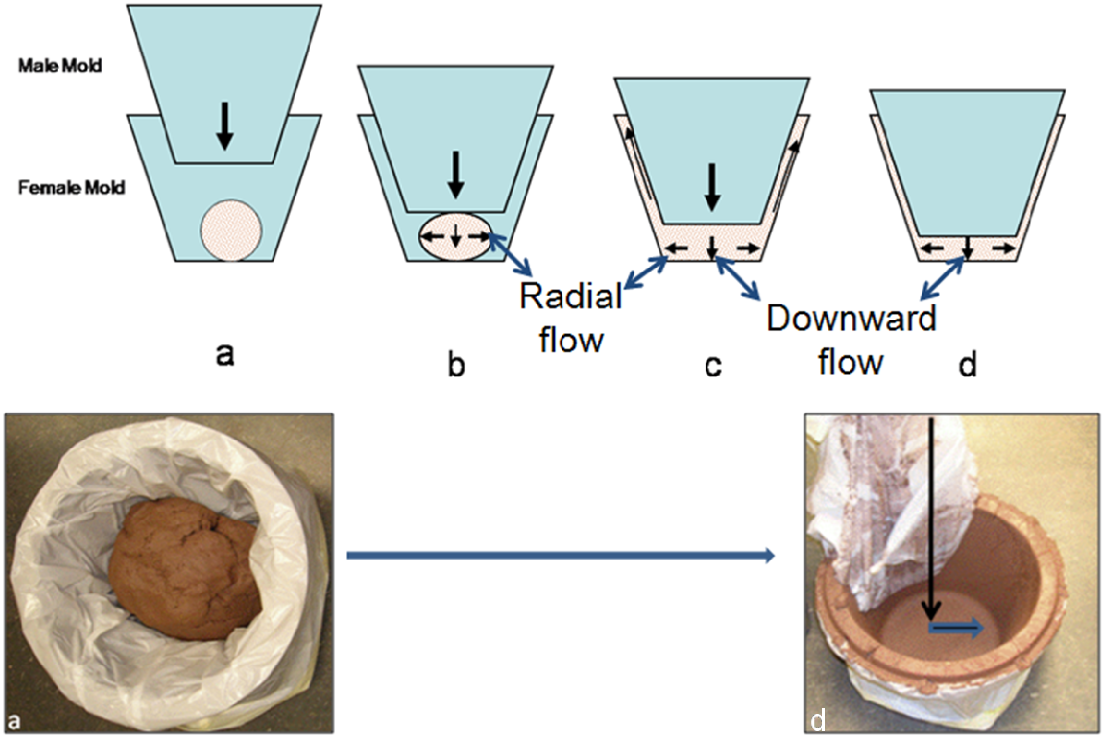

Figure 4.2: Two-dimensional representation of the development of the V-shaped filter during the axial press forming operation.

The greenwares were kept at room temperature to air dry. The time taken for drying varied from 5-8 days for each of the different constituent, volume ratio mixtures tested here. After drying, the greenwares were oven dried at 100°C for an hour in order to ensure complete moisture removal. The greenwares were then pre-heated to approximately 500°C for 3 hrs in an electric kiln [Cuff, 1996]. They were then slowly cooled to room temperature and then reheated in the electric kiln at the Ceramic Arts Department, Princeton University, Princeton, NJ, USA, to approximately 955°C. The rate of heating was augmented from 50°C/hr to 100°C/hr, after the kiln had reached 200°C. This process was sustained for 5 hrs at 955°C afterwhich the furnace was cooled to room temperature [Plappally, 2009].

The above mentioned liquid phase sintering process helps control the porosity of the resulting distinct ceramic material or porous clay ceramicware (PCCW) [Kang, 2005]. Behavior of clay ceramic while firing is controlled by mineral composition, particle size, environment and volumetric constituents, particularly decomposable minerals which help produce light-weight ceramicware, added other than clay [Henry, 1955]. Sawdust, with an 80% Oak and 20% Spanish cedar sawdust composition, decomposes to charcoal which helps in the production of the light-weight frustum-shaped PCCW. An C-S (Volume fraction of clay – Volume fraction of sawdust) notation has been used to describe the PCCW and their corresponding experiments.

Frustum shaped (PCCW) ceramicwares with the mixtures of 75-25, 65-35 and 50-50 had average porosities of 36.5%, 41% and 47%, respectively [Plappally, 2009]. Porosity measurements for the clay ceramicwares were performed using the mercury intrusion porosimetry MIP device, Micrometrics® Autopore III: 9400 digital analyzer. The procedure for using this analyzer has been discussed briefly in Chapter 3. The physical appearance, micro-structure and mechanical properties for clay ceramics manufactured at 955°C will be constant and unique. This is due to the fact that porosity and mechanical properties are highly influenced by temperature and other external factors [Nandi, 2008, Papargyris, 1996, Kang, 2005].

4.3 Experimental procedure

4.3.1 Material Analysis

The densities of the PCCWs were determined using a densitometer, Accupyc® 1330 V2. 01 (PRISM Labs, Princeton University). The increase in volume of sawdust augmented ceramic porosity, while ceramic density decreased with sawdust addition. These results were not observed in earlier experiments with inorganic material additives to clay [Papargyri, 2006]. These new experimental PCCW density data, Y (gm/cc), with configurations of 75-25, 65-35 and 50-50 were pooled together to analyze variation with corresponding volume fraction of sawdust, X. A transformation was applied to the predictor variable X. The new transformation $Z=X/Y$ is expressed as:

$$\ln Z = \ln a + b \ln X \tag{3}$$

The density Y was determined with a coefficient of determination $R^2 = 99.98\%$, and a standard deviation, s = 0.0031 in Eq. 4, as a function of volume fraction of sawdust X as:

$$Z = e^{-1} X^{0.998} \tag{4}$$

The experimental density values of the PCCWs as shown in Fig. 4.3 were in agreement and within the range of approximately 2-4 Mg/m^3 [Ashby, 1999, Soboyejo, W, 2003].

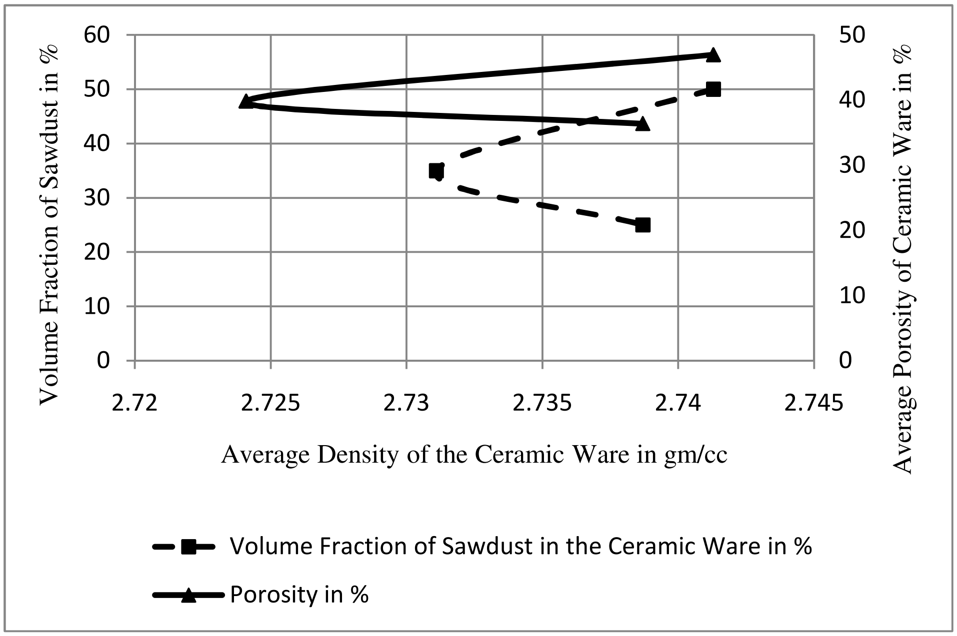

Figure 4.3: The average density as a function of average porosity (%) for three different specimens [(●) ,(■) and (▲)] of clay ceramicware containing 25, 35 and 50% sawdust by volume, respectively.

4.3.2 Bending and Compression Experiments

4.3.2.1 Bending Test

Fracture toughness tests were performed on single-edge notched bend (SENB) specimens with a thickness B, of approximately 12.6 mm, approximate width W of 12.6 mm, and approximate Span, S of 75 mm. All specimens were uniformly processed with a uniform notch to width ratio (a_o/W) of approximatelyt 0.2519 as shown in Fig. 4.4, for the SENB tests.

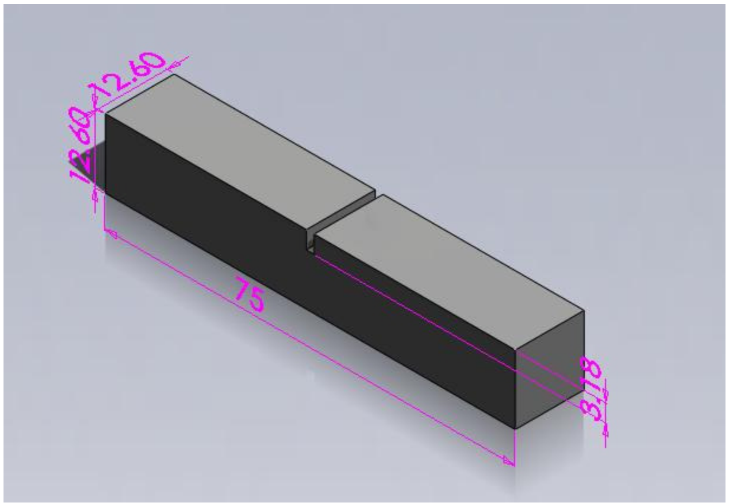

Figure 4.4: The CW SENB specimen with appropriate dimensions.

The SENB specimens with notch at center, and parallel to the circumference of the circular ceramic base of the PCCWs, are T-specimens, as shown in Fig.4.5. To evaluate anisotropic mechanical property variations, similar SENB specimens with notch at the center, and carved perpendicular to the circumference of the circular ceramic base were prepared as shown in Fig. 4.5.

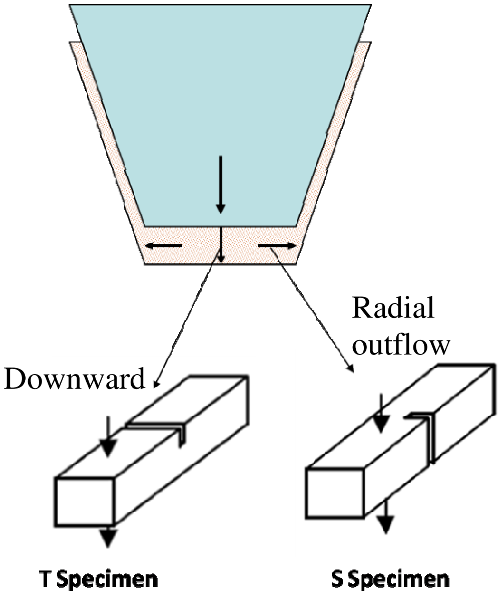

Figure 4.5: Characterization of specimens according to the two basic transport modes of wet clay-sawdust suspensions noticed during axial press forming operation as shown in Fig. 4.2.

The S-specimen (Fig. 4.5) is used to study the influence of the radial outward plastic flow (Fig.4.2) of clay-sawdust suspensions during the axial press forming process of the circular base of the ceramic. The specimen notches were carved out with a low speed diamond saw, and tests conducted under three-point loading with a span, S, of approximately 75 mm.

For each of the T- and S- specimens, three separate samples were taken from each of the 75-25, 65-35 and 50-50 configurations. Fracture toughness (K_{Ic}) tests and flexural (F_s) tests were conducted on both specimen types. An Instron Model 5848 electro-mechanical micro-testing machine (Instron®, Canton, MA) equipped with a 500-N load cell was used for fracture toughness testing. The tests were performed under load control at a monotonic loading with a ramp rate of 0.1 N/sec. Monotonic refers to a constant loading rate applied on the specimens with no reversals from initiation of the load application to a final fracture as per ASTM E-399-90 Mode I fracture test standard [Savastano, 2006, Suresh, 1991]. The tests were conducted at a room temperature of 298 K and a relative humidity of ~ 40-50%.

Both load and displacement data were recorded digitally using data acquisition software, LabView (Version 8.6 Software, NI Inc, Austin, TX). The loads corresponding to specimen failure were used for the calculation of the Mode I fracture toughness K_{Ic} . Fracture toughness, K_{Ic}, under the linear elastic fracture mechanics is given by [Soboyejo, 2003],

$$K_{Ic} = f\left(a/W\right)\left(PS/BW^{1.5}\right) \tag{5}$$

where W is the width, of the clay composite ceramic ware (CW) specimen (Fig.4.3) in the direction of the applied stress σ. Here the geometric and compliance function $f(a/W)$, for a single edge notched specimen is given by [Soboyejo, 2003],

$$f(a/W) = f(\chi) = (3\sqrt{\chi}/2(1+2\chi)((1-\chi)^{1.5}))\left[1.99 - \chi\left((1-\chi)(2.15 - 3.93\chi + 2.7\chi 2\right.\right. \tag{6}$$

From the three point bend tests, the flexural strength F_s of both the T-specimen and S-specimen were calculated using the formulae, $F_s = 3PS/2WB^2$, where P is the fracture load, W is the Width, B is the thickness and S is the span of the SENB specimen.

4.3.2.2 Compressive Stress Testing

The Compressive strength tests conforming to ASTM 1358 code standards. The specimens were of 12.6mm × 12mm × 35mm dimension. These were cut from the circular base of the frustum shaped PCCWs. Specimens were tested for compressive failure at a sustained static loading rate P of $0.1\mathrm{N} \cdot \mathrm{s}{-1}$ applied parallel to the 35mm span of the specimens. The tests were carried out on an Instron model 8872 electro-mechanical (Instron®, Canton, MA) machine with a 10KN load maximum capacity. The tests were conducted at a room temperature of 298° K and a relative humidity of 40-50%.

4.4 Theoretical Development

Linear elastic fracture mechanics parameters are influenced by compositional changes in the clay matrix and ceramic micro-structure [Buresch, 1985]. The fracture toughness K_{Ic} of a material is dependent on the basic composition, micro-structure of material and loading rate [Soboyejo, 2003]. For K_{Ic} to be accepted as a material property, a strict criterion is that K_{Ic} values are independent of specimen thickness [Soboyejo, 2003]. This criterion is checked using a Pearson correlation analysis between K_{Ic} and thickness B of the CW specimens. The statistical correlation between K_{Ic} and thickness B is -0.142 with a probability p value of 0.915 at a 95% confidence level. Hence, confirming that the fracture toughness is independent of specimen thickness, conforming to plain strain conditions [Soboyejo, 2003]. The value of K_{Ic} is found to increase with increase in volume fraction of clay used in the manufacturing. This is confirmed in Fig. 4.6, which plots a non-linear decrease in fracture toughness, K_{Ic}, with an increase in the volumetric content of sawdust.

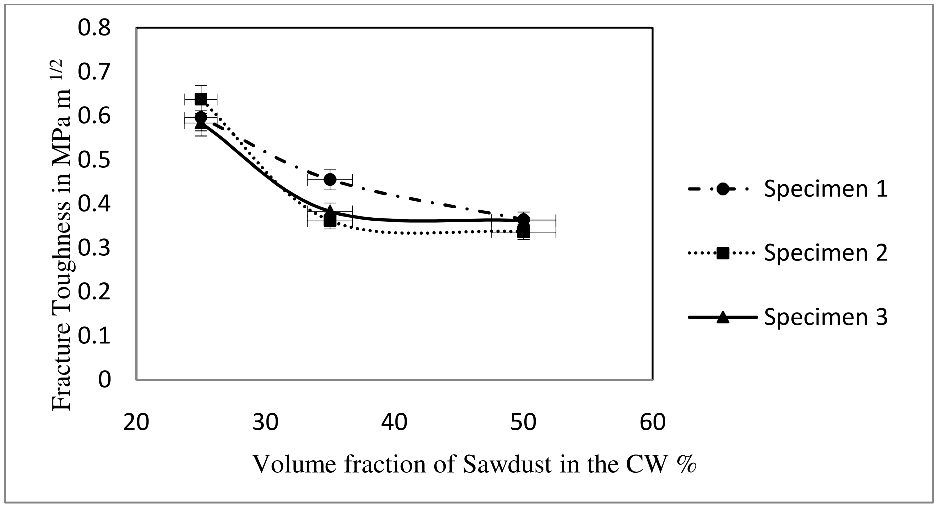

Figure. 4.6: Fracture toughness as a function of the volume fraction of sawdust for the three different T-specimens [(●) ,(■) and (Δ)] of clay ceramicware contaning 25, 35 and 50% sawdust by volume, respectively.

Experimental fracture toughness K_{Ic} values were within the range of 0.1 - 2.0 MPam$^{1/2}$ [Soboyejo, 2003, Wegst, 2004]. The strength of clay ceramics is a random statistical quantity depending upon the volume fraction of sawdust and stress state [Papargyri, 2003]. Hence the property of fracture toughness K_{Ic} of the ceramic material is a contribution of multiple parameters. These multiple parameters are step-by-step manifestations of raw material, micro-structural properties and loading effects influencing the final ceramic being tested. So, fracture toughness K_{Ic}, can be expressed mathematically as follows:

$$Y_i/Y_{i-1} = X_i^{b_i} \text{ for } i = 1, 2, 3 \tag{7}$$

Every step in the strength development (response variable Y) is influenced by a transfer function X_i^{bi} . X_i for i= 1, 2, 3, are the multiple predictor random variables discussed here: b_i is a constant applicable at each step i=1, 2, 3 (Benjamin et al., 1970). There are wide deviations in the fracture toughness values during the experiments on each of the three samples in the three compositions of the ceramicware specimens, as illustrated in Fig. 4.6. The value for K_{Ic} for the T-specimen CW decreased non-linearly with increase in sawdust volume fraction. This nonlinear behavior is simulated with a stochastic step function from step 0 to step 1 for one of the predictor variables as shown in Eq. 8:

$$Y_1/Y_0 = X_1^{b_1} \text{ for } i = 1, 2,3 \tag{8}$$

Where: $Y_0 = a$, initial value of the stochastic process model, and is a model constant in this problem.

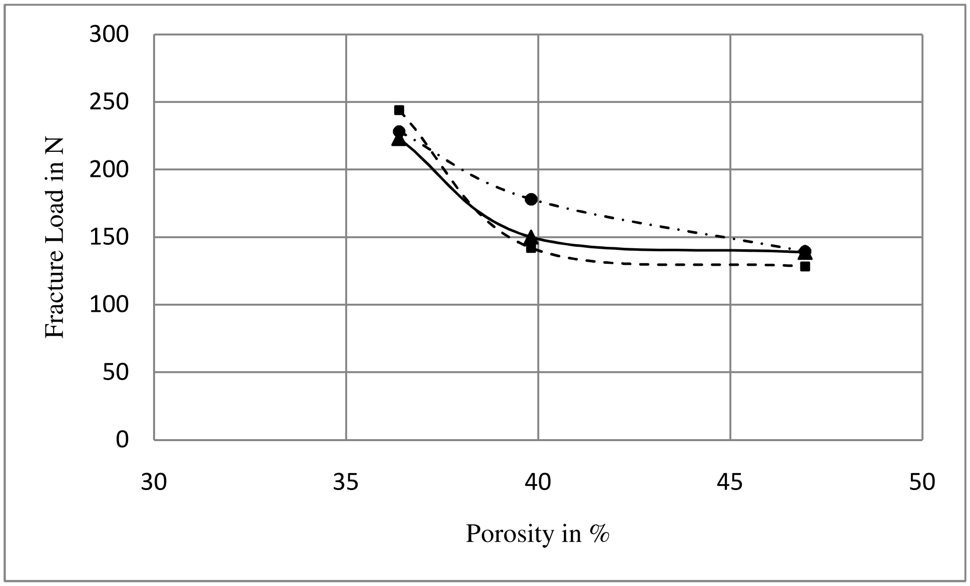

Figure 4.7: Fracture load as a function of porosity (%) for the three different T-specimens [(●) ,(■) and (▲)] of clay ceramicware contaning 25, 35 and 50% sawdust by volume, respectively.

Predictor random variables of X_i for i =1, 2, 3 are fracture load, volume fraction of sawdust, and porosity, respectively. From Fig. 4.7, it is found that predictor variables are very much dependent on each other. The load at which fracture occurred decreased with an increase in sawdust content. Similarly porosity contributed to a decrease in the ability of the specimens to bear larger loads. This confirms the fact that the strength of the heterogeneous material is dependent on micro-structural properties [Papargyris, 1996]. The observed nonlinear nature of fracture toughness can be written in the lognormal form as:

$$Y = aX_1^{b_1} X_2^{b_2} X_3^{b_3} = a\prod_{i=1}^{3} X_i^{b_i} \tag{9}$$

The above formulation, where a, b_1, b_2 and b_3 are model constants, presents the predictor variables in the multiplicative form.

Since the predictor random variables have different dimensions, Eq. 9 can be mathematically reformulated as:

$$Y = Y_o \prod_{i-1}^{3} (X_i / X_{io})^{b_i} \qquad (10)$$

$$a = Y_o \left\{ \prod_{i=1}^{k} X_i^{b_i} \right\}^{-1} \qquad (11)$$

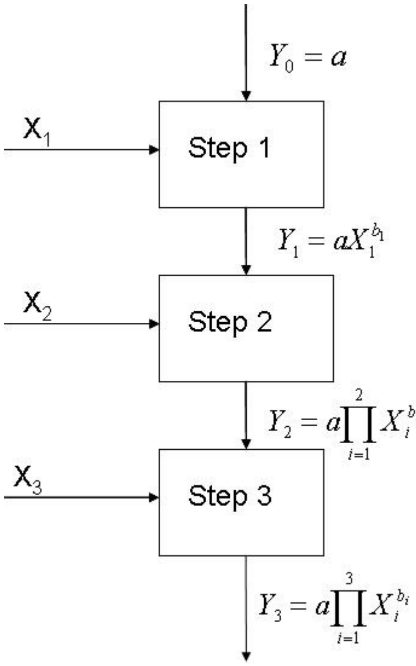

Figure. 4.8. Stochastic lognormal development with variables $X_i^{b_i}$.

Where: X_{io} is any reference constant with the same units as X_i, and Y_o is the intial value of the clay–sawdust mixture at the beginning of the PCCW manufacturing process. This helps in achieving dimensional similarity.

In order to carry out regression-based variance analysis, linearization of the multiplicative non-linear formulation is required [Soboyejo, 1965, Benjamin and Cornell, 1970]. In order to achieve this important result, the natural logarithm is taken on both sides of Eq. 9, and expressed as:

$$y_i = \ln Y_i = \ln a + \sum_{i=1}^{3} b_i \ln X_i = e^a x_1^{b_1} x_2^{b_2} x_3^{b_3} \qquad (12)$$

4.5 Results and Discussion

From Fig. 4.3, the density of the PCCWs is found to increase with an increase in sawdust content. If the density exceeds that of its manufacturing constituents, it should be noted that, channel pores may have been formed within the clay ceramic system as a result of phase transformations or chemical interactions between the constituents in the system [Mosin, 1996]. These materials with channels will help in effective water filtration [Mosin, 1996].

4.5.1 T-Notch Specimen Strength Analysis

Fig. 4.6 illustrates the variation of fracture toughness, K_{Ic} in T-specimens with respect to the volume fraction of sawdust. Each value on the plot represents K_{Ic} values from three different SENB tests on specimens of a specific composition. The toughness values vary for each of the three T-specimens 1, 2 and 3 tested in each of the volume fractions as $K_{Ic50\text{-}50\text{-}T} < K_{Ic65\text{-}35\text{-}T} < K_{Ic75\text{-}25\text{-}T}$, and K_{Ic} decreases with increasing porosity. With increase in porosity there is a decrease in surface area on which the monotonous load is distributed on the pre-crack zone. This is due to the fact that in this two-phase porous ceramic material, the phase in the pores has zero stiffness [Davidge, 1979]. Fig. 4.7 illustrates a similar trend as followed by fracture load (P) for each of the three specimens 1, 2 and 3 tested in each of the volume fractions, and varies as $P_{50\text{-}50\text{-}T} < P_{65\text{-}35\text{-}T} < P_{75\text{-}25\text{-}T}$.

Predictor Variables\ Model Coefficients	$\boxed{\overline{a}}$	$\boxed{\overline{b_1}}$	$\boxed{\overline{b_2}}$	$\boxed{\overline{b_3}}$	$\boxed{R^2}$	**S**
X_1	-6.02	1.01			99.8	0.012
X_2	-6.66	1.09	0.0687		99.9	0.0098
X_3	-5.19	1	0.103	-0.305	100	0.00199

Table 4.1: Improvement in the prediction of K_{Ic} for T-specimens using variables X_1, fracture load, X_2, volume fraction of sawdust in the ceramicware, in % by volume of the raw materials, and X_3, porosity of the ceramicware in %, using: $y_i = \ln a + \sum_{i=1}^{3} b_i \ln X_i$.

Table 4.1 shows the step-by-step improvement in prediction with increasing predictor variables and decreasing error S of the model. Correlations exist between the predictor variables X_1, X_2 and X_3 as shown in Table 4.2. It should be noted that regression is performed with only statistically independent variables.

Predictor Variables	X_1	X_2	X_3
X_1	1	-0.924	-0.959
X_2	-0.924	1	0.933
X_3	-0.959	0.933	1

Table 4.2: Statistical correlation between predictor variables: X_1, fracture load, X_2, volume fraction of sawdust in the ceramicware in % by volume of the raw materials, and X_3, porosity of the ceramicware in %.

The correlated predictor matrix column elements lnX_i= $\overline{X_{\iota,J}}$ with correlation coefficient $\rho_{X_{i,j}}$ as tabulated in Table 4. 2 are linearly transformed into mathematically uncorrelated variables V_i=$\overline{V_{\iota,J}}$and scaled as [Soboyejo, 2001]:

$$\frac{\left(\left[\bar{X}_{i,j}-\mu_{\overline{X_{\iota,J}}}\right]\right)}{\sigma_{\overline{X_{\iota,J}}}} = [\bar{T}]\left[\frac{\left[\overline{V_{\iota,J}}\right]}{\sqrt{\lambda_{i,j}}}\right] \tag{13}$$

The left hand side of Eq. 13 are standard normal variates of the predictor variables ln$X_{i,j}$ for $i \neq j$ and i=1, 2, 3 having zero mean and unit standard deviation. Here $\mu_{\overline{X_{\iota,J}}}$ and $\sigma_{\overline{X_{\iota,J}}}$ are the parameters of a normal distribution. The multivariate analysis approach used through Eq. 13 is elaborated in the Appendix 1.

Using Eq. 13, the stochastic multi-variate, lognormal model for fracture toughness of the T-specimens is given by Eq. 14 (MINITAB 15, Minitab Inc.PA):

$$y = e^{-5.19} V_1^{-0.618} V_2^{-0.852} V_3^{-0.0123} \tag{14}$$

where the coefficient of the third variable is larger than the coefficient of the first variable and the second variable. Here the coefficient of determination and standard deviation in prediction of Eq.14 is same as tabulated in Table 4.1. This confirms the fact that the micro-tructure of the heterogeneous porous ceramic material has a maximum influence on strength. The model coefficients b_1 = -0.618, b_2 = -0.852 and b_3 = -0.0123 in Eq. 14 confirms the non-linear decrease in fracture toughness with increasing magnitude of the predictor variables.

A log-transformation reduces the magnitude of the actual raw data thus reducing the mean and error values associated with the raw data. Errors due to the predictor variables x_1, x_2 and x_3 influence errors in the response variable, y_i in log-transformed Eq. 12. Hence, a non-parametric variance analysis using a first order second moment method is conducted to study the influence of the errors in the predictor variables on the response corresponding to the T-specimen [Haldhar and Mahadevan, 2000, Schneider, 1997, Soboyejo, 1968].

$$Var(y) \approx (b_1)^2 Var(x_1) + (b_2)^2 Var(x_2) + (b_3)^2 Var(x_3) \qquad (15)$$

$$0.0644 \approx 0.0627 \qquad +0.0009 \qquad +0.00106$$

This confirms a pronounced influence of micro-tructure on the fracture toughness than quantitative amount of materials required for manufacture. A comparative study of variances needs to be performed to confirm these effects of predictor variables between different specimens and for any specific specimen composition. Table 4.3 enumerates this comparative study.

Two way ANOVA test						
Sources of Error	Sum of Squares	DOF	Mean Squares	f-Statistic	p value	R^2
Between CWs	13.418	2	6.709	122.56	0.00	91.08
Within a CW	1.313	24	0.054			
Total	14.732	26				

Table 4.3: Analysis of variance between predictor variables: X_1, fracture load, X_2, volume fraction of sawdust in the ceramicware in % by volume of the raw materials, and X_3, porosity of the ceramicware in % towards prediction of K_{Ic} for the CWs.

From Table 4.3, the large value of the f-statistic = 122 indicates a significant difference in mean fracture toughness values between the three CW variants studied in this problem, and not much variations within a particular CW. Since the variance ratio tabulated for a level of significance of $\alpha = 0.05$, $f_{2,24:0.05} = 3.40$ is less than the calculated f-statistic = 122, the hypothesis that mean K_{Ic} values for all T-specimens tested are equal is rejected. These points at a very low p-value which is the probability of obtaining a K_{Ic} value equal or more extreme than what has been plotted in Fig 4.6. This confirms the significant difference in the fracture toughness K_{Ic} of the three CWs tested here.

4.5.2 S-Notch Specimen Strength Analysis

Fig. 4.9 illustrates the variation of fracture toughness K_{Ic} for the S-specimen with respect to the volume fraction of sawdust in the CW in %. Each value on the plot represents fracture toughness values of three different SENB tests on specimens of a specific distinct compositions. It is found that K_{Ic} values of both T- and S- specimens were in complete agreement and fell within the ranges of K_{Ic} for porous ceramics [Ashby, 1999, Soboyejo, 2003].

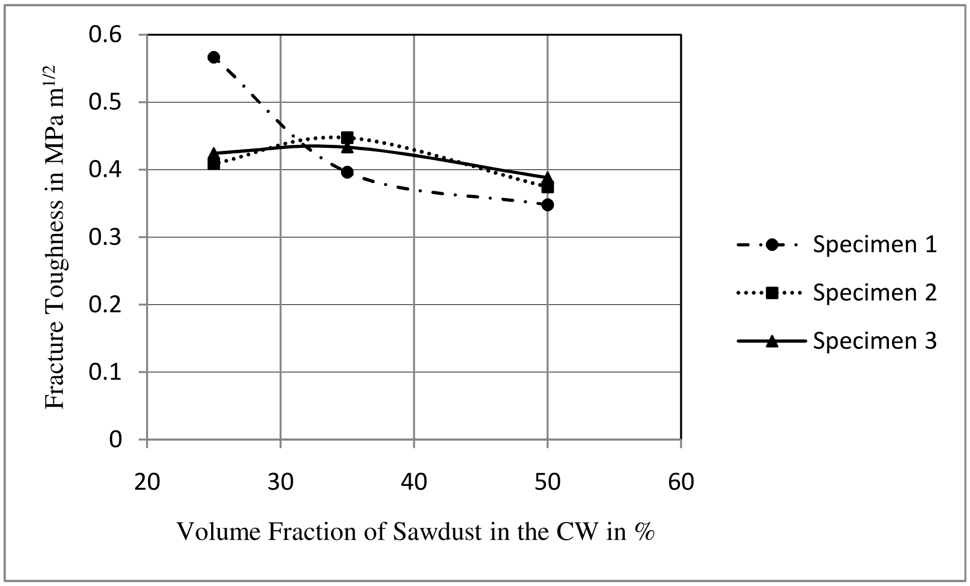

Figure.4.9: Fracture toughness as a function of volume fraction of sawdust for the three different S-specimens [(●) ,(■) and (Δ)] of clay ceramicware contaning 25, 35 and 50% sawdust by volume, respectively.

The fracture toughness values vary for each of the three S-specimens 1, 2 and 3 tested in each of the volume fractions $K_{Ic50\text{-}50\text{-}S} < K_{Ic65\text{-}35\text{-}S} < K_{Ic75\text{-}25\text{-}S}$ and thus it is seen that K_{Ic} for S-specimens decreases with increasing porosity.

The stochastic, multi-variate lognormal model for fracture toughness of the S-specimens is given by Eq. 14 (MINITAB 15, Minitab Inc.PA):

$$y = e^{-0.875} V_1^{0.114} V_2^{-0.0729} V_3^{-0.119} \qquad (16)$$

This confirms the negative influence of porosity on the tensile fracture toughness. It is seen that $b_1>b_2>b_3$, which enumerates also the least influence of porosity of the toughness of the S-specimen.

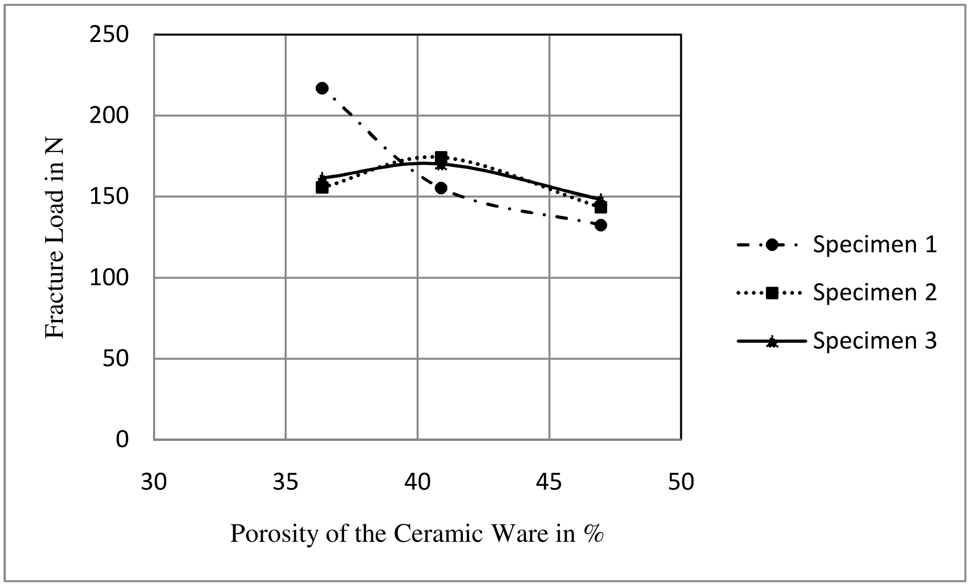

Figure. 4.10: The fracture load (P) as a function of porosity (%) for the three different S-specimens [(●) ,(■) and (▲)] of clay ceramicware contaning 25, 35 and 50% sawdust by volume, respectively

Fig. 4.10 illustrates fracture load as a function of porosity. The fracture load P of a S-specimen varies as $P_{50\text{-}50\text{-}S} < P_{65\text{-}35\text{-}S} < P_{75\text{-}25\text{-}S}$. From Figs. 4.6, 4.7, 4.8 and 4.9, it is noted that $K_{Ic\ 75\text{-}25\text{-}T} > K_{Ic\ 75\text{-}25\text{-}S}$, $P_{75\text{-}25\text{-}T} > P_{75\text{-}25\text{-}S}$, $K_{Ic\ 65\text{-}35\text{-}T} < K_{Ic\ 65\text{-}35\text{-}S}$ *and* $K_{Ic\ 50\text{-}50\text{-}T} < K_{Ic\ 50\text{-}50\text{-}S}$, and , $P_{65\text{-}35\text{-}T} < P_{65\text{-}35\text{-}S}$ *and* $P_{50\text{-}50\text{-}T} < P_{50\text{-}50\text{-}S}$.

There will be a specific percentage by volume of sawdust above 25% volume fraction of sawdust beyond which fracture toughness of T-specimen is reduced in comparison to the S-specimen. This behavior may be due to variations in crack shielding effects due to changes in the volumetric distribution of sawdust within clay body and its orientation.

The strength of S-specimens is larger than for the T-specimens, which may be due to plastic flow of raw materials while manufacturing as enumerated in Fig 4.2. This would mean a possible directional alignment of sawdust taking place due to the plastic flow in S-specimens affecting crack growth. This strength development phenomenon increases with increase in volume fraction of sawdust in the CWs.

4.5.3 Flexural Strength Calculations

It is enumerated from Fig. 4.11 that flexural strength F_s of the T-specimen is less than for the S-specimen for the each of the samples from the 65-35 and 50-50 CWs. It is also seen that the magnitude of the flexural strength values for the CWs for both T- and S-specimens were close to the findings reported in the literature [Nandi, 2008, Lee, 2008]. This agreement is clearly seen in Fig. 4.12. It is also enumerated that flexural strength for the T- and S-specimens in the 75-25 CW varies as $F_{s\ 75\text{-}25\text{-}T} > F_{s\ 75\text{-}25\text{-}S}$.

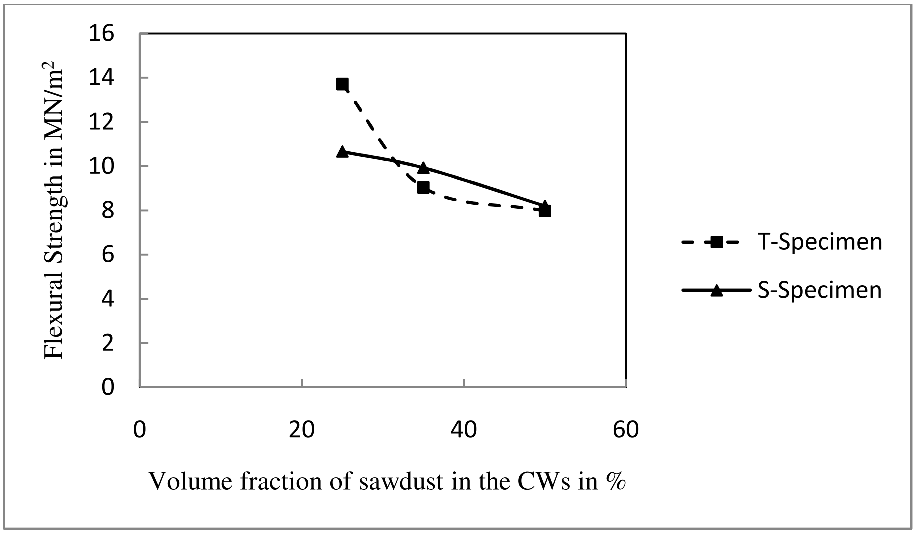

Figure. 4.11: Flexural strength as a function of volume fraction of sawdust for T-and S-specimens [(■) and (▲)] of clay ceramicware.

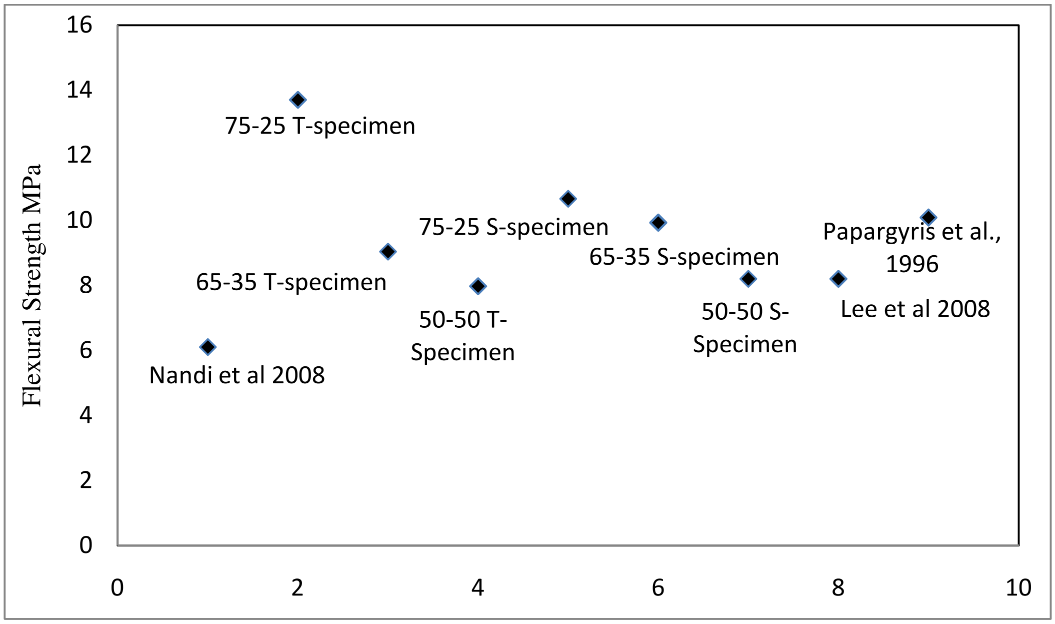

Figure. 4.12: Comparative study of range of flexural strength of the fired kaolin based ceramics tested herein and similar studies conducted by different authors for a sintering temperature of 950^{o}C [Nandi et al, 2008, Lee et al, 2008, Papargyris et al, 1996].

The behavior of flexural strength of the 75-25 CWs is similar to the behavior of their respective fracture strength. This reiterates the possible crack shielding effects due to changes in the volumetric distribution of sawdust within the clay body and its orientation.

4.5.4 Compressive Strength Analysis

Compressive load tests conforming to ASTM C1358 were performed on the clay ceramic material specimens. The average compressive strength is found to decrease linearly with increasing porosity. A straight line equation for the compressive strength of the clay CWs as a function of porosity has been proposed in agreement to that put forward by Liete et al. (2001) for uniaxial compression strength measurements on porous materials. This is represented in Fig.4.13.

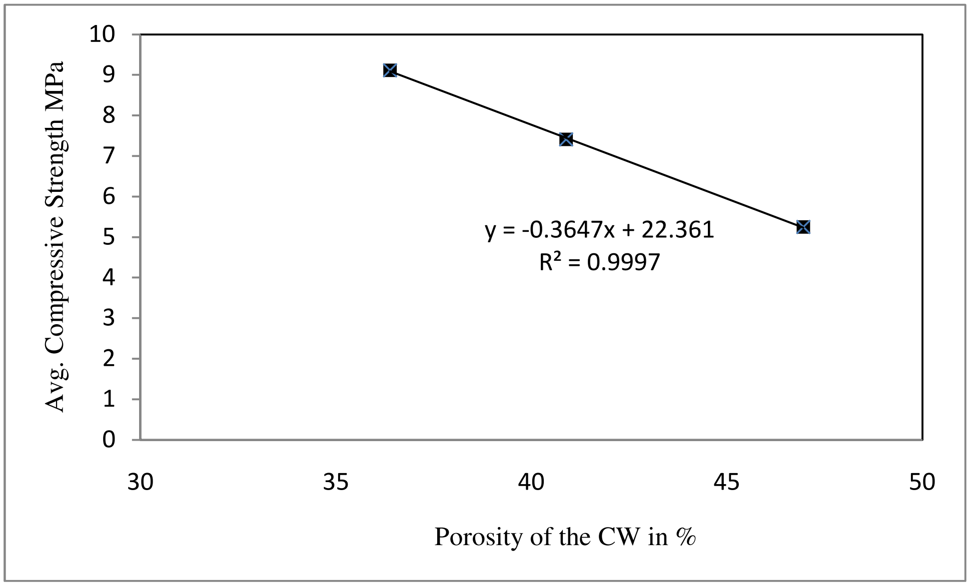

Figure 4.13: The average compressive strength as a function of porosity for the three different specimens (■)of clay ceramicware contaning 25, 35 and 50% sawdust by volume, respectively.

The compressive failure load is found to increase non-linearly with increase in volume fraction of clay in the CWs as shown in Fig. 4.14. The observed nonlinear nature of compressive stress may be written in the lognormal form as,

$$Y = aX_1^{b_1} X_2^{b_2} = a\prod_{i=1}^{2} X_i^{b_i} \qquad (17)$$

Linearization of the multiplicative non-linear formulation in Eq. 17 is achieved by log-transformation of X_i. This can be expressed as [Soboyejo, 1965, Benjamin and Cornell, 1970]:

$$y = \ln Y_i = \ln a + \sum_{i=1}^{2} b_i \ln X_i = e^a x_1^{b_1} x_2^{b_2} \qquad (18)$$

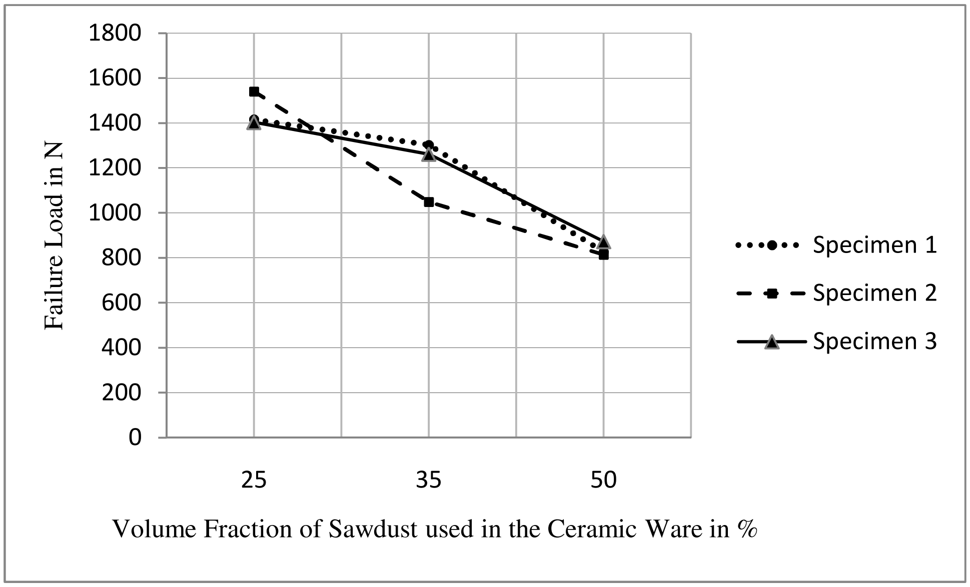

Figure. 4.14: The compressive failure load as a function of volume fraction of sawdust for the three different specimens [(•) ,(■) and (▲)] of clay ceramicware contaning 25, 35 and 50% sawdust by volume, respectively.

Table 4.4 illustrates the step-by-step prediction of uni-axial compressive stress as a function of multiple parameters as shown in Eq. 18.

Predictor Variables\Model Coefficients	$\bar{a}$	$\overline{b_1}$	$\overline{b_2}$	R^2	**S**
X_1	-5.04	0.995		99.8	0.0097
X_2	-4.49	0.940	-0.0477	99.9	0.0092

Table 4.4: Improvement in prediction of uniaxial compressive stress using variables failure load X_1 , volume fraction of sawdust in the CW X_2, using $Y = a\prod_{i=1}^{k=2} X_i^{b_i}$

The volume fraction of sawdust, X_2 and compressive fracture load, X_1, used for the prediction of compressive stress are interdependent. The Pearson correlation coefficient for the two variables is -0.959. Multivariate analysis, as enumerated in Eq 13 and explained in Appendix 1, is used to remove this interdependence. Then the stochastic regression model for compressive stress with independent predictor variables is given by:

$$Y = e^a V_1^{-0.177} V_2^{-0.163} \qquad (19)$$

where a = 1.96. From Eq. 17, X_2 has a larger influence on the compressive strength of the CWs as compared to X_1. Both X_1 and X_2 have a negative effect on the compressive strength of the ceramicware.

Analysis of variance based on multi-parameter regression methods is used to verify the individual errors of the random variables. The parameter *Var (y)* is the error in the prediction of the response variable, y in Eq. 18. *Var (x_1)* and *Var (x_2)* are variance or error expected in individual predictor variables x_1 and x_2 as given in Eq. 18. From empirical data analysis and calculations we have:

$$Var(y) \approx (b_1)^2 Var(x_1) + (b_2)^2 Var(x_2) \tag{20}$$

$$0.0623 \approx (0.940)^2 [0.0629] + (-0.0477)^2 [0.090] \tag{21}$$

$$0.0623 \approx 0.05557 + 0.000204 \tag{22}$$

It is important to note from Eq. 24 that $((b_1)^2\ Var(x_1))$ is much larger, hence variability arising from X_2 is much smaller than X_1.

4.6 Conclusions

Experiments were conducted on porous clay ceramic material sintered at 950°C with known compositions of clay and sawdust. The interactions between the volumetric, physical and material variables influencing strength of clay ceramic materials have been studied. The major inferences of this study are summarized below.

1. In brittle clay ceramic materials with organic material constituents like sawdust, there is a nonlinear relationship between the average density of the ceramic material and volumetric amount of organic constituents.
2. Fracture toughness development in brittle clay ceramic materials is dependent on the direction of plastic flow that occurs during forming stages of manufacture.
3. Simple multiplicative models make it easier to predict the compressive strength of clay composite ceramic bodies at ambient temperatures.
4. The volume fraction of sawdust used for manufacturing the clay composite ceramic material plays a defining role in predicting its porosity, density and mechanical properties. It should be known that temperature has a major influencing role on the above mentioned properties, and hence a specific study has been carried out only on clay ceramics manufactured at a sintering temperature of approximately 950°C and tested at ambient temperatures.

Models similar to those derived in this chapter to predict strength development may be used for prediction of strength in similar ceramics manufactured at different temperatures.

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Chapter 5: Stochastic Multivariate Regression Based Approach Using Thermodynamics of Irreversible Processes Applied to Leaching Effects in Clay Ceramic Ware

Abstract

Abstract: This chapter provides a brief discussion on the chemical dissolution taking place at the initial stages of gravity-driven filtration through ceramic filters. Positive pH variation has been used as a means to study leaching in porous clay ceramicware (PCCW) filters. These filters were locally manufactured in different places using clay and sawdust specific to the respective locations. Uniform mixing of these materials with water, molding and sintering processes were used in the manufacture of the PCCW filters. The results of water percolation tests for these filters elaborated by different authors were used for this study. Three different cases are studied. The first case analyzed filtrate pH as a function of filtration time and type of material used in PCCW manufacture. Multivariate regression hypothesis based on the Onasager assumption forms the basis of the model formulation predicting the rise of filtrate pH in the second case using thermal and electro-kinetic parameters. The third case uses thermal, electro-kinetic and time as parameters for the prediction of a positive increase in the pH of the PCCW filtrate.

5.1 Introduction

Transport through nano-porous structures have helped in the filtration and purification of water. These ceramic, nano-porous structures are basically made of clays. Water flow through ceramics will be affected by changes in micro-pore structure [Churaev, 1990; Derjaguin et al, 1987]. The compositional changes of the material surface due to dissolution also affect water flow [Churaev, 1990; Derjaguin et al, 1987]. Water flow through porous media may be modeled by continuum models, capillary bundle models, pore-scale network models, and other numerical stochastic models [Philip, 1986, Sochi, 2009, Trussell et al, 1999]. A model solution is still non-existent connecting flow with material as well as compositional aspects of the fluid varying with time.

Halem (2006) elaborated on the influence of dissolution of the chemical constituents of porous clay ceramicware (PCCW) on pH with flow occurring over a period of time. Figs. 5.1, 5.2 and 5.3 represent the experiments related to long-term analysis of pH with percolation of water through clay ceramic filters from Ghana, Cambodia and Nicaragua, respectively.

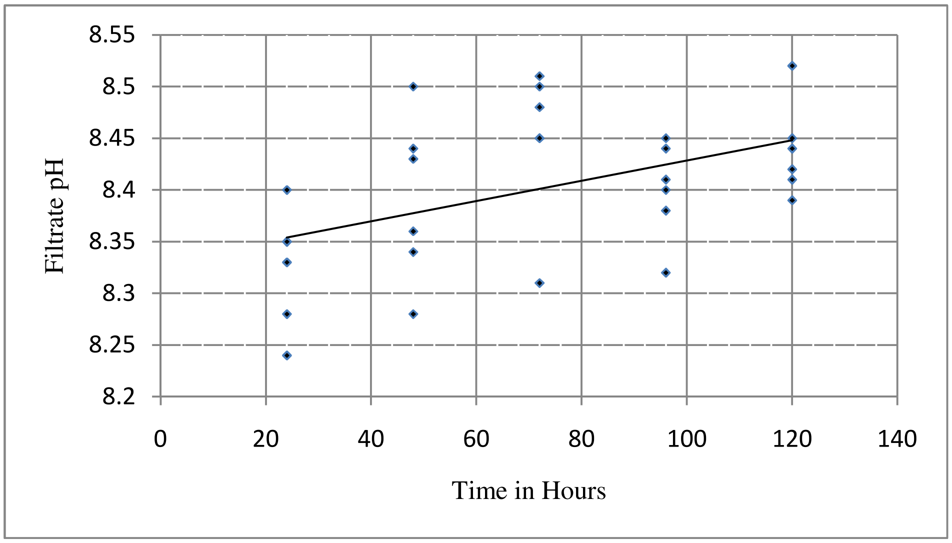

Figure 5.1: Filtrate pH in PCCW filters from Ghana [Halem, 2006].

Dissolved oxygen increases on an average of 1.3 mg/L after filtration, due to increased air contact as the water drips from the filter [Lantagne, 2001]. Lantagne showed that an increase of 0.8 pH units was seen on post-filtration due to alkalinity of the clay-based ceramic [Lantagne, 2001]. Halem experimentally verified the increase in pH [Halem, 2006].

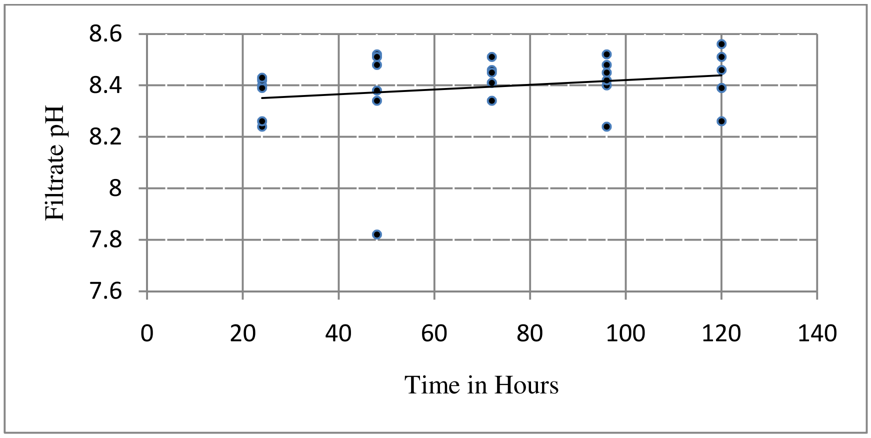

Figure 5.2: Filtrate pH in PCCW filters from Cambodia [Halem, 2006].

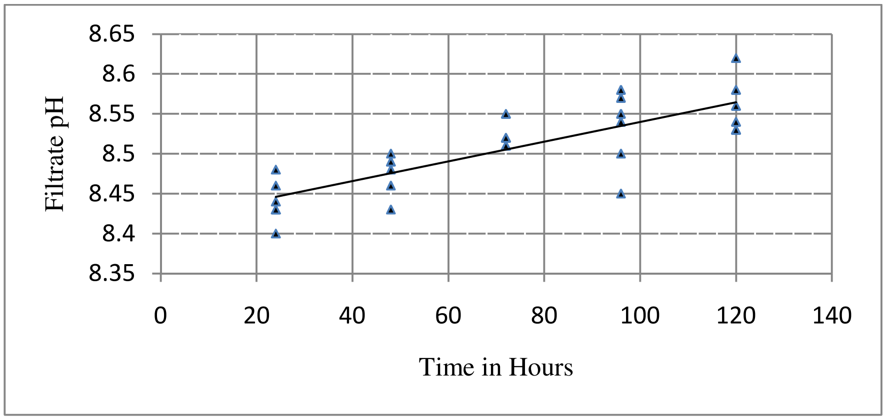

Figure 5.3: Filtrate pH in PCCW filters from Nicaragua [Halem, 2006].

Flow through clay-based substances occurs due to non-equilibrium conditions between the porous ceramic structural interfacial volume and fluid volume [Bachmann et al, 2002, Marmur, 1992a]. Non-equilibrium conditions suggest irreversible processes which are responsible for an increase in entropy [Onasager, 1931]. It is to be noted that the entropy production rate in any system can be expressed in terms of mass or molar fluxes [Onasager, 1931, Kjelstrup, et al, 2001, Prigogine, 1977]. The experimental findings of oxygen dissolution by Lantagne (2001) and increase in alkalinity in filtrate during filtration by Halem (2006) represent examples of irreversible processes. When dealing with flow through ceramic porous membranes there is a need to account for flow under a concentration gradient, ΔC, flow under an electric potential, ΔE, flow under a temperature gradient, ΔT, and flow under a pressure gradient, ΔP [Sonune, et al. 2004].

5.1.1 Temperature Influence on Flow through Porous Media

The flow of liquid through ceramic pores due to temperature differences is defined as thermo-osmosis [Churaev, 2000, Trussell, et al, 1999]. The ceramicware manufactured and discussed herein is a porous body filled with water. The porousware can be considered as series of discrete connected reservoirs in contact between which constant pressure and temperature differences are maintained. The solvent flux, S_f, with units of mole per unit area per second can be given as:

$$S_f \approx a\nabla\mu_p + b_1\nabla T/T \tag{1}$$

Where: a is a constant related to the filtering capacity of the porous media, and b_1 is a constant related to the flow due to a temperature gradient, ∇T. Here $\nabla\mu_p = -m_v\nabla P$ and $a = k/\eta m_v$, where m_v is the molar volume of the water (liquid), ∇P is the pressure gradient across the thickness of the porous media, η is the viscosity of water (liquid) and k is a filtration coefficient. Eq. 1 can be rewritten as:

$$S_f \approx -k\nabla P/\eta + b_1\nabla T/T \tag{2}$$

The second term on the right hand side of Eq. 2 is equal to the mass rate of flow through the porous media due to a temperature difference on both sides of the porous media. As the porosity of the media decreases, capillary pressures drop [Tiller and Tsai, 1986]. An increase in temperature and porosity of ceramic materials results in decrease in thermo-osmotic velocity [Churaev, 2000]. Thermo-osmotic velocity can be written with the knowledge that enthalpy change (ΔH) (positive in the case of hydrophilic surfaces, for example clay ceramics) due to the temperature gradient, ∇T, porosity p and thickness of the porous media h as:

$$V_{th} = -ph^2 \Delta H \nabla T / 2\eta m_v T = (\chi / m_v) \nabla T / T \qquad (3)$$

For nano-porous clay ceramics, the χ values are negative and thermo-osmotic flows are towards the lower temperature side of the porous structure [Keijzer et al, 1999]. Rejection of salts is not dependent on thickness of porous media, h, and can be modeled with an effective pore radius and ionic charge density [Bowen et al, 2002]. Fig 5.4 presents a comparative difference in the temperatures of the influent water and effluent filtrate of the PCCW filters tested at Delft by Halem (2006).

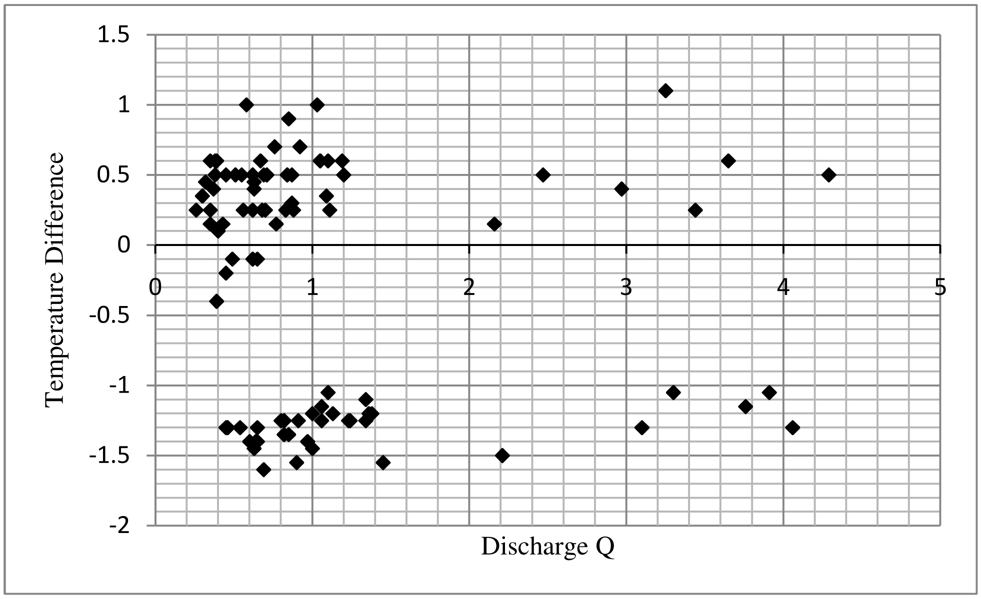

Figure 5.4: Temperature gradient with respect to discharge Q from the PCCW filters irrespective of the location of manufacture (Ghana, Cambodia, Nicaragua as per Halem, 2006)

5.1.2 Influence of Ionic Concentration on Flow through Porous Media

Clay ceramics have electro-kinetic properties when in contact with water [Worrall, 1986]. Charge develops on clay ceramic compounds in water basically due to diffusion. When water flows through any porous media it generates a streaming current, which is directly proportional to water conductivity [Paillat et al, 2001]. Fig 5.5 depicts the variation of pH with change in the volumetric content of the materials used in the manufacture of similar porous ceramic filters as discussed by Halem (2009). The pH variation of the filtrates in the ceramic vessel indicated the possible dissolution of reflux ions present in clay, such as K, Na, Mg, Ca Al, etc. [Halem, 2009]. They promote alkalinity in the pure water used in the experiment.

Fig. 5.5 shows the variation of pH as a function of volume content of sawdust used in manufacturing the clay-ceramic filters [Plappally et al, 2009]. The volume of sawdust used to manufacture the clay ceramic is a minimal order statistical property [Ganapathysubramanium et al, 2007].

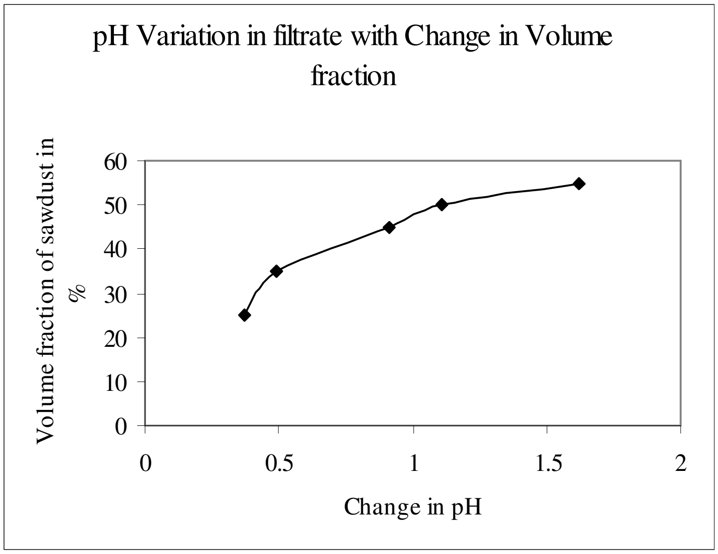

Figure 5.5: Variation of the pH in the filtrate with changes in volume fraction.

From Fig. 5.5, the change in pH (using a Model D8611, Barnstead/Thermolyne, Hampton, NH) follows a rising trend with volume fraction of sawdust used in manufacturing a specific filter. The non-linear change in pH in the filtrate volume, Y_i, is a random function of the sawdust, X_i for $i= 1$.

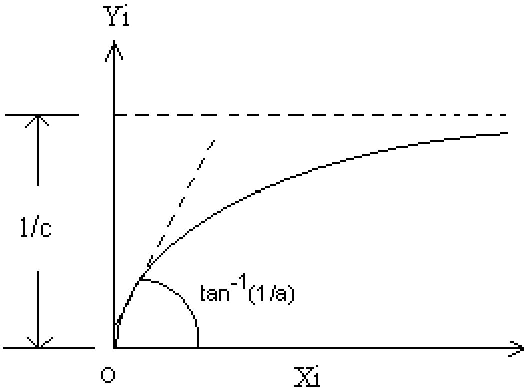

Figure 5.6: Slope and intercept of development of Y_i as a function of X_i similar to Fig. 5.5.

The increase in pH of the filtrate, Y_i, as mathematically elaborated in Fig. 5.6, may be the result of dissolution of chemical ions from the filter. From Fig. 5.6, X_i is transformed keeping in mind that this will not change the original pH behavior [Plappally et al, 2009; Tiller and Hsyung, 1991], as:

$$Y = \frac{X_i}{a + \sum_{i=1}^{k} c_i X_i} \quad \text{for i = 1,2 ……k} \tag{4}$$

Eq. 4 can be rewritten as:

$$Z_i = \frac{X_i}{Y} = a + \sum_{i=1}^{k} c_i X_i \quad \text{for } i = 1, 2,\text{……k} \tag{5}$$

where a, is a constant related to the the ceramic material coming in contact with percolating pure water.

The coefficient of the predictor variables X_i for i= 1, 2, 3…..k are c_1, c_2 …c_k which provide information about the importance of the influence of each of the predictor variables on response Z_i in this case [Bulmer (1957)]. The regression model from the Y data as plotted in Fig. 5.5 may be modeled as shown in Table 5.1.

Predictor Variables\Model Coefficients	a	c_1	S	R^2
X_1	1.07	0.105	0.0015	0.982

Table 5.1: The regression model of pH as a function of volume fraction of sawdust X_1.

With an decrease in clay content by volume in the filter, the compaction of the sawdust and clay may decrease. This may result in clay loosely adhering to the sawdust which may enhance the surface area of the clays to easily come in contact with water, and this may result in enhanced dissolution of reflux ions in the clay. This can be seen from the model in Table 5.1 and also from Fig 5.5.

5.1.3 Capillary Pore influences on Flow through Porous Media

Diffusion depends on the degree of hydration of the cation attached to the ceramic surface and its concentration [Tadros, 1992]. Exchange of ions and concentration of cations near the ceramic clay surface induce zeta-potential variations [Worrall, 1986]. Thus, in porous ceramics, the liquid medium near the clay surfaces tend to maintain equilibrium dispersion of chemical composition [Churaev, 2000]. Clay suspensions in water are colloidal in nature and can be characterized with Brownian and diffusion-based motion, with which the clay constituent ionic particles tend to collide. While colliding, some particles join together to form macromolecules or flocs. These collisions are dependent upon four major forces acting on the particles.

First, short range forces act on all solid surfaces. Secondly, electrostatic forces are based on the electrical double layer or charges of colliding particles. Third, there is a rapid increase in the repulsive energy with increasing closeness of two colloidal particles [Tadros, 1992]. Finally there are Van der Waals forces which increase with decreasing distance between two particles. So in order to bring the charged particles closer, the above mentioned forces have to be overcome.

The agglomeration of ions near the surface of the clay ceramic in water is basically governed by a layer of ions. Positive cations tend to strongly cling near to the surface forming the stern layer. This positive density of charges σ induces a potential ψ_{sl} on the surface as:

$$\sigma = \epsilon_0 \kappa \psi_{sl}/4\pi \qquad (6)$$

where ϵ_0 is the static dielectric permittivity of water, an $1/\kappa$ is distance at which potential dips e times or Debye radius [Churaev, 2000]. There is a potential difference and it is seen that there is a dip in electric potentials in this stern layer due to high density of cations. This dip of electric potential at the surface distance x from the porous surface ψ_s to that in the stern layer ψ_{sl}which is be represented as [Tadros, 1992]:

$$\psi(x) = \psi_{sl} \exp{(-\kappa x)} \qquad (7)$$

Capillary osmosis is associated with a dense layer of ions near the ceramic surface. Since solid liquid separation is the major process for which these ceramic wares are used, the degree of filtration plays a major part in defining transport influenced by concentration differences ∇C [Churaev, 2000]. Hence solvent flux is experessed as:

$$S_f \approx -k\nabla P/\eta + b_2 \nabla \mathrm{C} \qquad (8)$$

where b_2 defines the velocity of the capillary flow due to concentration gradient, ∇C, which is a measure of the electrical conductivity. The presence of dissolved ions may cause deviations from Darcy's law in porous media due to capillary osmosis [Churaev, 2000].

5.1.4 Dissolved Ions influencing Flow through Porous Media

Electrical conductivity (micro-siemens/cm) quantifies the amount or concentration of total dissolved ions in the water, and is determined by the chemistry of the influent water, material properties of the PCCW, microbial metabolism if any, and temperature effects. Fig. 5.7 illustrates hat specific conductance or electrical conductivity of water improves with increase in temperature. Specific conductance indirectly measures the presence of dissolved ions. The amount and mobility of ions determines conductivity. These ions are Cl^-, NO_3^-, SO_4^{2-}, PO_4^{3-}, Na^+, Mg^{2+}, Ca^{2+}, and Fe^{2+}/Fe^{3+} and can be used to indicate impure water. The ionic strength greatly influences microbial activity [Paulsen et al, 1997, Morales et al, 2007].

While dispersion takes place in non-rigid porous media, like clay ceramics, there is slow displacement of surface particles bound to the clay ceramics. This may cause blockage of the pores reducing hydraulic conductivity or permeability [Soma et al, 1995], which suggests structural changes which may occur during flow that causes porosity variation with time [West et al, 1999]. When the particles on the surface get dispersed in the direction of flow the porous structure is deformed.

Due to the heterogeneous weak ionic/covalent composition of the porous ceramic ware, this phenomenon becomes pronounced with time [Philip, 1986, Zhu et al, 2000]. This process will increase the potential difference within the clay ceramicware and atmosphere.

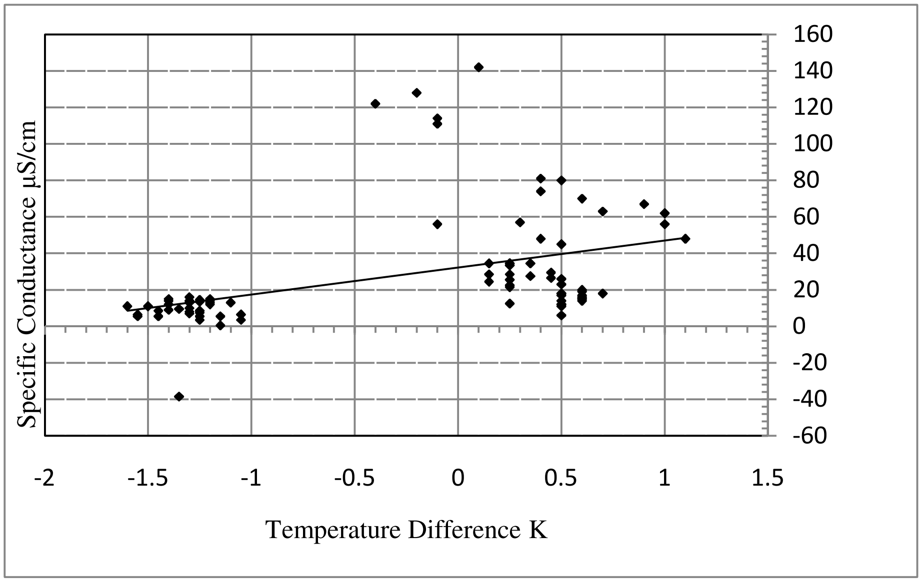

Figure 5.7: The variation of the concentration of total dissolved ions with temperature.

This potential difference will induce flux of solute through the porous media, which can be stated by the equation given below for dilute solutions under negligible thermal influence, as:

$$S_f \approx -k\nabla P/\eta + b_3\nabla \mathrm{E} \qquad (9)$$

where $b_3 = -\kappa/\sigma\eta m_v$ defines the velocity of exchange of charge between molecules across the ceramicware or porous media [Abramson et al, 2007]. This velocity, which is also known as streaming potential $j = pL/A_s$, is directly proportional to the change in pressure or head of liquid to be percolating through the porous media [Rastogi et al, 1970]. Here L is the average perimeter of the pores and A_s is the average cross-sectional area of the pores [Churaev, 2000].

If we consider thermal influences in Eq. 9, the capillary pressure difference in clay ceramic porous media will be greatly influenced by temperature changes in the environment rather than just by the temperature of influent water [Bachmann et al, 2002]. This influence may be due to three major mechanisms: the wetting contact angle incremental changes for liquid (probably with some organic acid like humic acid in our case [She et al, 1998]) over the heterogeneous irregular surfaces of the clay ceramic; the concentration effecting interfacial tension in this two-phase system; and enthalpy changes due to wetting of clay ceramic [Bachmann et al, 2002, She et al, 1998]. Permeability is directly proportional to change in fluid pressure, therefore Darcy's formulation on porous media has a permeability term which is a pressure dependent term [Buermann, 1999].

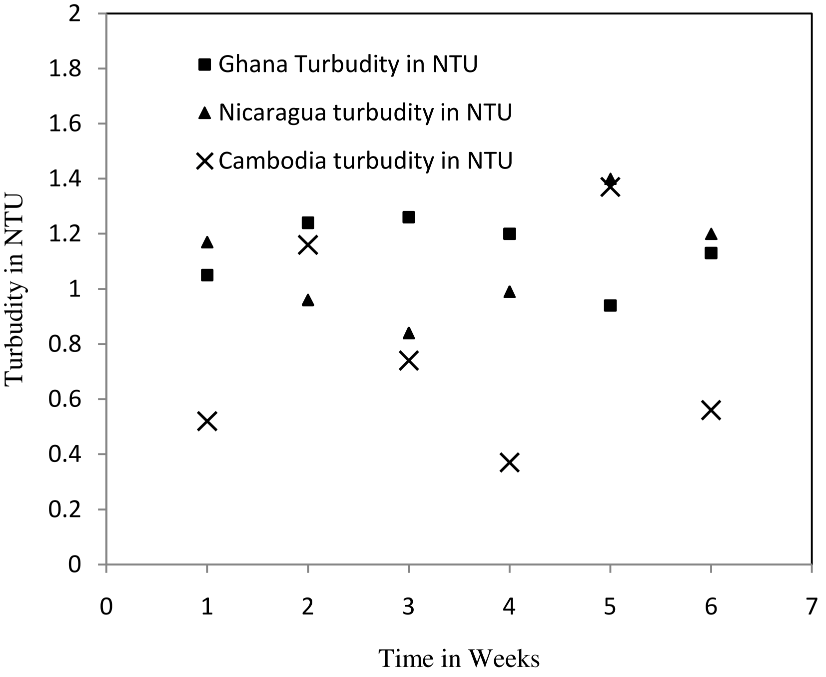

Figure 5.8: Turbidity variations with respect to time from the PCCW filters irrespective of the location of manufacture (Ghana, Cambodia, Nicaragua from Halem, 2006].

The electro-osmotic pressure within the porous media is linearly proportional to the potential difference between the thicknesses of the ceramic porous media [Srivastava et al, 1977]. It is to be noted that electro-osmosis is a major factor in exchange of materials within cellular organisms in water. The cellular organism in unfiltered water, such as E.coli and other microbes (biological colloids), affect the velocity of exchange of charge between molecules [Bradford, et al 2007; Churaev, 2000; Kjelstrup et al, 2005]. Turbudity (Nephelometric units or NTU) defines the degree of microbial, organic/inorganic chemicals, as well as colloidal contents of water [EPA 1997].

Based o the literature above, the solute flux through porous ceramic clay structures can be written in the framework of thermodynamically irreversible processes, as:

$$S_f \approx -k\nabla P/\eta + b_1\nabla T/T + b_2\nabla \mathrm{C} + b_3\nabla \mathrm{E} \qquad (10)$$

Thus, non-equilibrium thermodynamic conditions proposed by Onasager are very much satisfied by clay and water-based suspensions [Srivastava et al, 1972].

From Figs. 5.1, 5.2, 5.3, the pH of the effluent filtrate increases with time. From the literature, the measure of change in potentials at influent and effluent may be influenced by several thermodynamic parameters, such as temperature, electrical conductivity, mass or volumetric flux-force systems, and changes in concentration. This indicates a stochastic multi-parameter process behind the development of positive pH gradient.

Multiple predictor random variables, whose successive values depend on the preceding value, nfluence this stochastic development [Haldar et al, 2000]. Stochastic multi-predictor processes have been proposed to predict the leaching characteristics of the clay ceramics filter during filtration, thus proposing a possible coupling of chemical dissolution and transport. Based on the models developed here, the errors or variances have been examined and quantified.

5.2 Experimental Data [Halem, 2006] on Filtration Parameters under Ambient Temperature and Pressure Conditions and Methodology

The dependence of flow through PCCW filters on manufacturing material composition and processing have been studied. The experiments were conducted at the University of Delft by Van D Halem. The data of the predictor variables responsible for the development of change in pH between the water filter influent and effluent (ΔpH) are referred from those experiments. The author of this technical document acknowledges the retrieval of the data from Halem (2006).

The raw data are then modified according to the requirements of a stochastic regression analysis. It is noteworthy to mention that the original tests were conducted in an elaborate way, discussed by Halem (2006). A multitude of variables were documented for both influent and effluent chemical thermodynamic parameters for water percolating through frustum shaped PCCW filters. The filters in Ghana, Cambodia and Nicaragua were manufactured with locally available clay and sawdust, mixed at 50% clay (C) and 50% sawdust (S) volumetric ratios. The general local manufacturing procedure of the 50-50 (C-S) PCCWs was followed for all cases [Halem, 2006, Lantagne, 2001].

Weekly tests measured the change in turbidity, pH, temperature and electrical conductivity between the influent water and the filtrate. These measurements are shown in Figs. 5.1, 5.2, 5.3 and 5.4, respectively, based on weekly experiments on a series of 24 filters from all locations mentioned above [Halem, 2006, Halem et al, 2007, Halem et al, 2009]. Ceramic discharge was measured simultaneously with all weekly experiments and is plotted in Fig 5.4 as a function of temperature difference. The three types of PCCW filters were Type 1, Type 2 and Type 3 from Ghana, Cambodia and Nicaragua, respectively.

Since we are interested in the positive change in pH with time between influent and the filtrate from 50-50 PCCW filters, theoretical multivariate modeling is conducted in two case studies.

In case 1, a comprehensive experimental data analysis is conducted to predict filtrate pH as a function of time X_1 for filtration through the PCCW filter and type of material X_2.

Case 2 predicts ΔpH with multiple chemical thermodynamic predictor parameters that influence heterogeneous porous media flow. Case 2 simulates relation between flow of water and changes in environmental variables. Case 3 is an extension of Case 2 over a time span.

5.3 Theoretical Development

5.3.1 Case I: Time and Material influence on pH

The pH data as a function of time was provided by Halem and co-authors in 2009. These data have been reviewed and confirmed for model suitability. The data for Type 1, Type 2 and Type 3 PCCW filters were pooled separately. Each type had six filters and filtrate pH data were collected for 12 weeks. For our calculations, 5 weeks of data were considered, to skip the changes in data during maintenance operations on the filters after 5 weeks as reported by Halem et al (2007; 2009). The purpose was to clearly study the behavior of gravity-driven flow through clay ceramics influenced by various environmental, material and thermodynamic parameters, respectively. Figs. 5.2, 5.3 and 5.4 show the data for Type 1 (Ghana), Type 2 (Cambodia) and Type 3 (Nicaragua) filters, respectively [Halem, 2009].

PCCWs	Model	a	c_1	R^2	S
Type 1	Z_1	0.00165	0.118	99.9	0.0026
Type 2	Z_2	0.00206	0.118	99.9	0.0053
Type 3	Z_3	0.00267	0.116	99.9	0.0016

Table 5.2: Stochastic regression model for pH as a function of time X_1following $Z_1 = a+\sum_{i=1}^{1} c_i X_i$

Table 5.2 represents the results of regression analysis which provides the model coefficients a and c_1. It also gives the coefficient of determination, R^2, and standard deviation of the model S.

When considering multi-material constituents, the chemical behavior of the location-specific clays has a great influence on the working of the filters [Kattamuri et al, 2005, Wilson et al, 1995]. In order to study influence of materials specific to Type 1, Type 2 and Type 3, respectively, Eq.5 is transformed to include a material specific parameter, M_{123}. The new model may be written as $Z = f(X_1, X_2)$, and

$$Z = m_0 + c_1 X_1 + c_2 M_{123} \tag{11}$$

where $M_{123} = a$, a is derived from Table 4.2 [Soboyejo et al, 2001]. The independent variables X_1 and M_{123} are pooled as per requirements for Eq. 11 as shown above.

	A	c_1	c_2	R^2	S
Z	0.355	0.00119	-0.167	99.9	0.0038

Table 5.3: Stochastic model of pH as a function of time and material property following

$Z_i = a + \sum_{i=1}^{2} c_i X_i$

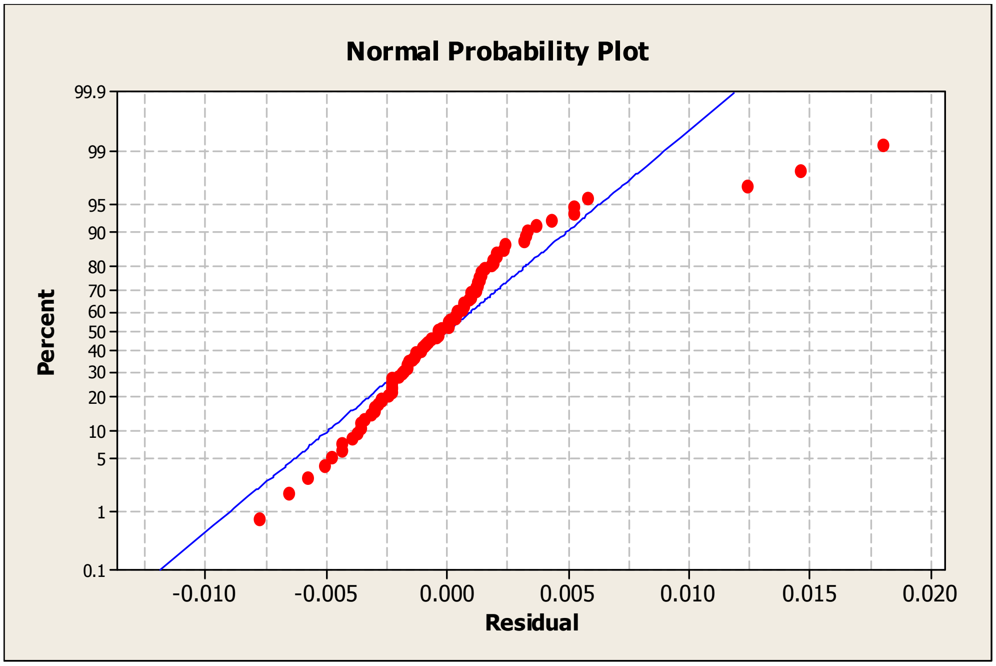

Figure 5.9: Normal probability plot of the transformed response variables Z [Minitab 15, 2009]

Fig. 5.9 plots points of the normally distributed Z value which confirms the goodness of prediction of the model represented in Eq. 11. From Table 5.3, coefficients c_1 and c_2 reflect the influences of the corresponding predictor variables. The values of model coefficients c_1 and c_2 show a greater influence of time on pH than the material property parameter. The material parameter M_{123} applies a negative effect on the development of pH.

5.3.2 Case II: Thermodynamic and ElectroKkinetic Influences on pH Change (ΔpH).

The influences of thermodynamic parameters on flux of ions through ceramic clay filter needs to be studied. The framework of non-equilibrium thermodynamics as elaborated by Onasager is used here for predicting leaching in clay ceramics. Leaching is predicted by an increasing pH value of the filtrate with respect to the influent water pH.

For the analysis, (ΔpH) instead of the volumetric quantity of solute flux S_f is taken as the response variable Y.

There is always a conducive environment for microbial (bio-colloids) bio-film growth on the water surface as well as surface of the porous ceramics [Klarman, 2009, Petrasch 2008]. Ionic transport phenomena near the bio-film boundary and total volume of fluid (water) influences turbidity [Paulsen et al, 1997]. The possible dissolution of exchangeable cations present in clay influence ionic transport. The other contributors for the changes in pH would be lignin and humic acids which may be formed in the process of saturation and filtration due to the presence of sawdust. Hence change in turbidity plays an important role in quantifying the organic and inorganic dissolution mentioned above.

Influent fluid chemistry influences filtration characteristics of colloidal removal by straining [Bradford et al, 2007]. Hence the next predictor variable, $X_{2,}$ is the change in turbidity between the influent water and effluent filtrate.

Velocity in comparison to it thermal component influences porous media flow the most [Mansfield et al, 2000]. Flow rate Q will help to vary pH with time., and th change in pH of the water to be filtered may change the hydraulic conductivity or permeability of the clay ceramic filters and vice versa [Santiwong et al, 2008]. Ergun's relations for porous media flow do not conform to Reynolds numbers greater than 20 [Hellstrom et al, 2006]. The above discussion reiterates the importance of flow rate Q as a major parameter for consideration in this analysis.

An increase in the variation of pH is seen with an increase in the temperature difference in Fig. 5.10. This trend is common for all the experiments performed by Halem (2006).

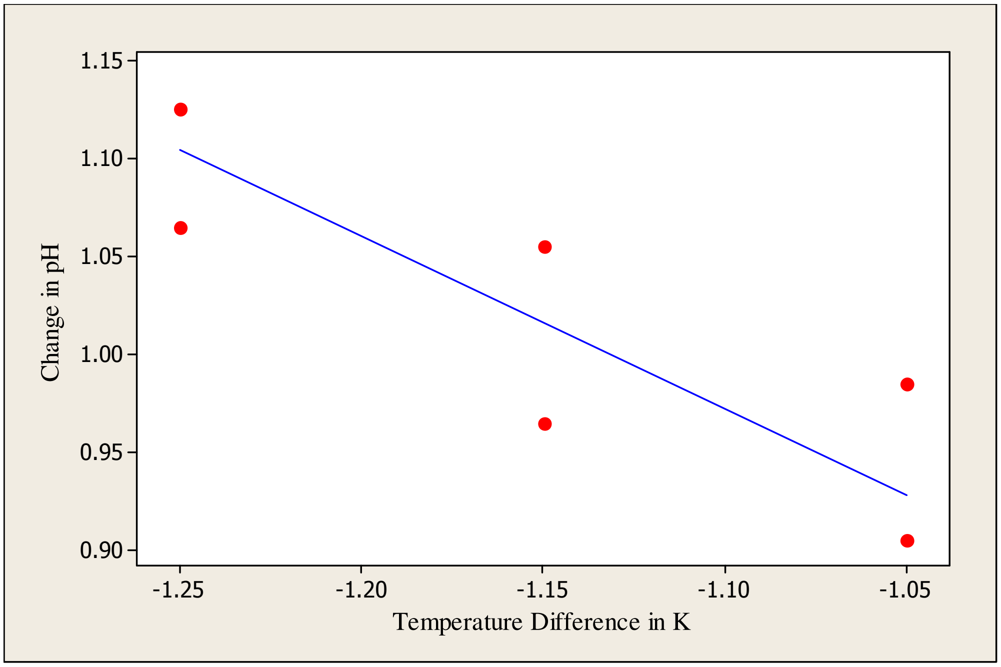

Figure 5.10: ΔpH as a function of temperature difference between the influent water and effluent filtrate measured in week 2 for Type 1 filters [Minitab 15, 2009].

Thermal influence is the third variable X_3 considered. Presence of dissolved ions affects the flow rate of the filters. Flow rate Q variations with time influences electrical conductivity. Hence the fourth predictor variable X_4 is the change in electrical conductivity between the influent and effluent filtrate from the clay ceramic filters.

Predictor Variables	X_1	X_2	X_3	X_4
X_1	1	0.28	0.92	-0.318
X_2	0.28	1	0.319	-0.473
X_3	0.92	0.319	1	-0.37
X_4	-0.318	-0.473	-0.37	1

Table 5.4: Correlation coefficient $\rho_{X_{i,j}}$ between the predictor random variables, difference in turbidity of the influent and filtrate X_1, filtrate discharge rate X_2, temperature difference

of the influent and the filtrate X_3 and difference in electrical conductivity of the filtrate and the influent X_4.

Table 5.4 confirms how the reciprocal relationships expounded by Onasager is true in case of predictor random variables, difference in turbidity of the influent and filtrate X_1, discharge rate X_2, temperature difference of the influent and the filtrate X_3 and difference in electrical conductivity of the filtrate and the influent X_4 [Onasager, 1931]. This confirms the various transport processes occurring simultaneously during gravity-based filtration process are dependent on each other. A negative correlation is evidence that large values of the predictor variables X_i are associated with small values of the other counterpart predictor variables X_i [Bulmer, 1957]. Table 5.4 also correlates specific conductivity X_4 negatively to concentration effects X_1 due to turbidity as confirmed by Kohlrausch [Goodwin, 1899].

Continuing on the regression hypothesis by Onasager, material specific multivariate modeling of ΔpH $(=Y)$ is performed. Separate regression models have been depicted for each of the Type 1, Type 2 and Type 3 filters, respectively, with the different material and chemical properties of each type [Agyare, 2004, Hendrix et al, 2005, White et al, 2000].

A linear stochastic process can be expressed as:

$$Y=Y_i = a + b_1X_1 + b_2X_2 + \ldots\ldots\ldots\ldots b_kX_k \qquad (12)$$

where a = the initial value of the stochastic process $(a = Y_0)$; b_i for $i = 1, 2, 3\ldots k$ are model constants, Y is the response random variable, X_i for $i = 1, 2, 3\ldots.k$ are the predictor random variables as tabulated in Table 5.4. The stochastic linear model in Eq. 12 is diagrammatically shown in Fig. 5.11:

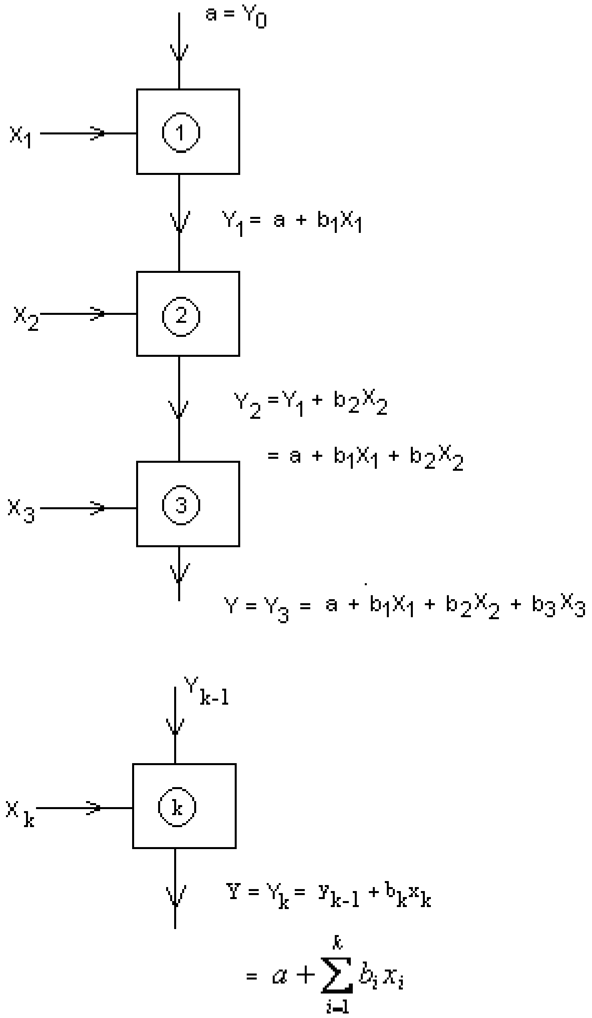

Figure 5.11: Linear stochastic model is formulated for multiple parameters [Soboyejo, 2000].

Since the predictor random variables have different dimensions, Eq. 12 can be mathematically reformulated as:

$$Y/Y_{io} = \sum_{i-1}^{k}(b_i X_i/X_{io})/Y_{io} \qquad (13)$$

where X_{io} and Y_{io} are any reference constants with the same units as X_i and Y_i, respectively. This enhances dimensional similarity, and also helps maintain the assumptions of dimensionless variables used by Onasager [Hemmer et al, 1996].

The correlated predictor matrix column elements in Table 5.4 with correlation coefficient $\rho_{X_{i,j}}$ are linearly transformed to independent variables V_i and scaled as [Haldhar et al, 2000, Krishnamoorthy, 2006]:

$$\frac{\left([\bar{X}_{i,j}-\mu_{X_{i,j}}]\right)}{\sigma_{\bar{X}_{i,j}}} = [\bar{T}]\left[\frac{[\overline{V_{i,j}}]}{\sqrt{\lambda_{i,j}}}\right] \qquad (14)$$

The left hand side of Eq. 14 are standard normal variates of the predictor variables $X_{i,j}$ for $i \neq j$ and $i=1, 2, 3$ having zero mean and unit standard deviation. Here $\mu_{\bar{X}_{i,j}}$ and $\sigma_{\bar{X}_{i,j}}$ are the parameters of a lognormal distribution. The correlation coefficient of the standard normal variates is now $\rho_{\overline{X_{1,j}}}$, where $\rho_{\overline{X_{i,j}}}$ = F parameter * $\rho_{X_{i,j}}$[Kiureghian, et al, 1985]. The F-parameter for Eq. 12 in the case of large (n>30) and normally distributed pH data is unity [Haldhar et al, 2000]. The general multivariate approach in Eq. 14 for removing the correlation between the predictor variables and making them independent is shown in Appendix. A.2.

Results of the multivariate linear stochastic formulations are shown in Tables 5.5, 5.6 and 5.7. Encouraging results are seen for Types 1 and 3 in comparison to Type 2 filters. This change may be due to material or chemical changes in the filters. For example, it is found that the porosity of Type 2 filters was around 45%, which may have affected porous flow processes to sustain as compared to the lesser porous Type 1 and 3 filters [Halem, 2007]. Similarly, tortuosity values of the Type 2 filters were the smallest in comparison to those of Types 1 and 3 [Halem, 2006]. Type 3 filters had the maximum tortuosity [Halem, 2006].

From Tables 5.5, 5.6 and 5.7, the model coefficients $\overline{b_i}$ for i= 1, 2, 3, 4, can enumerate independent effects of each variable in the positive change in pH.

Type 1	$\bar{a}$	$\overline{b_1}$	$\bar{b}_2$	$\bar{b}_3$	$\bar{b}_4$	R^2	S
Week1	0.565	0.0267	-0.0300	0.0313	-0.0465	99.7	0.00713350
Week 2	1.02	0.0452	-0.0168	-0.0373	-0.0100	97.6	0.0275562
Week 3	0.560	0.0348	0.0402	-0.0075	0.173	92	0.0461212
Week 4	1.03	0.0262	-0.00168	-0.0182	-0.179	100	0.00117285
Week 5	0.943	0.0164	-0.0295	0.0148	-0.001	90.7	0.0309775

Table 5.5: Multivariate model constants for Eq.12, predicting *ΔpH* (=*Y)* in Type 1 filters for 5 weeks.

Type 2	$\bar{a}$	$\bar{b}_1$	$\bar{b}_2$	$\bar{b}_3$	$\bar{b}_4$	R^2	S
Week 1	0.597	0.0269	0.018	-0.008	-0.0479	62.8	0.114706
Week 2	0.967	0.175	-0.0849	-0.107	-0.0116	100	0.00726960
Week 3	0.540	0.0048	-0.0037	-0.897	0.0462	81.7	0.0551140
Week 4	1.05	-0.0226	-0.014	0.231	0.0346	32.2	0.179065
Week 5	0.933	-0.0193	-0.0074	-0.405	0.0240	48.2	0.170811

Table 5.6: Multivariate model constants for Eq.12 predicting *ΔpH* (=*Y)* in Type 2 filters for 5 weeks.

Type 3	$\bar{a}$	$\bar{b}_1$	$\bar{b}_2$	$\bar{b}_3$	$\bar{b}_4$	R^2	S
Week 1	0.680	0.00195	-0.0133	-0.0091	0.0779	73	0.03204212
Week 2	1.10	0.00477	-0.00208	0.00665	-0.0455	94.3	0.0137660
Week 3	0.560	-0.0039	0.0039	0.0077	0.910	78.8	0.0750483
Week 4	1.17	-0.0243	0.0190	-0.0207	0.0183	81.5	0.0469021
Week 5	1.07	0.0107	0.0030	0.0015	0.0577	77.1	0.0377212

Table 5.7: Multivariate model constants for Eq.12, predicting *ΔpH* (=*Y)* in Type 3 filters for 5 weeks.

In Table 5.7, each week was separately modeled owing to continuous dissolution of flux element ions, which may give rise to new micro-structural parameters which may change the permeability of the clay ceramic filters [Philip, 1986, Stevenson, 1997, Zhu et al, 2000]. Flow rate from the ceramic filters X_2 has negative influence on *ΔpH (=Y).* This confirms Fick's Law, which states that flow rate may be written as $X_{2_i} = -D\nabla c_i$ for i = 1, 2, where c_i represents the concentration per unit volume and D is a constant [Onasager, 1945].

5.3.3 Case III: Extension of Case II Irrespective of Location and Material Composition.

From Table 5.2 and Eq. 12, pH increases with time. pH also behaves as shown in Fig. 5.6 and elaborated by Eq. 5. Eq. 5 can be rewritten as:

$$N = \frac{L_1}{\theta + m_1L_1 + m_2L_2 + m_3L_3 + m_4L_4 + m_5L_5} \tag{15}$$

And Eq. 15 can be reformulated as:

$$G = L_1/N \tag{16}$$

where N is the change in pH (ΔpH), L_1 is the difference in turbidity of the influent and filtrate (NTU), L_2 is the filtrate discharge rate (l/h), L_3 temperature difference of the influent and the filtrate in K, L_4 is the difference in electrical conductivity of the filtrate and the influent (S 10^{-8}/m), and L_5 is time in hours. Since the predictor random variables have different dimensions, Eq. 15 can be mathematically reformulated as

$$G/G_{io} = \sum_{i-1}^{k}(m_i\, L_i/L_{io})/G_{io} \tag{17}$$

where L_{io} and G_{io} are any reference constants with the same units as L_i and G, respectively. This helps in achieving dimensional similarity. Table 5.8 confirms that the predictor variables L_i are dependent on each other.

Predictor Variables	L_1	L_2	L_3	L_4	L_5
L_1	1	-0.139	0.471	0.874	-0.409
L_2	-0.139	1	-0.102	-0.223	-0.145
L_3	0.471	-0.102	1	0.421	0.257
L_4	0.874	-0.223	0.421	1	-0.421
L_5	-0.409	-0.145	0.257	-0.421	1

Table 5.8: Correlation coefficient $\rho_{L_{i,j}}$ between the predictor random variables L_1, L_2, L_3, L_4 and L_5.

The correlations in Table 5.8 confirms how the reciprocal relationships expounded by Onasager are true in the case of predictor random variables, differences in turbidity for the influent and filtrate L_1, discharge rate L_2, temperature differences for the influent and the filtrate L_3, and differences in electrical conductivity for the filtrate and the influent L_4 and time L_5. The various transport processes occurring simultaneously during gravity-based filtration processes through the PCCW filters is confirmed as being dependent on each other and with time.

The correlated predictor matrix column elements in Table 5.8 with correlation coefficient $\rho_{L_{i,j}}$are linearly transformed as independent variables V_i and scaled as [Haldhar et al, 2000, Krishnamoorthy, 2006]:

$$\frac{\left(\left[\bar{L}_{i,j}-\mu_{L_{i,j}}\right]\right)}{\sigma_{\bar{L}_{i,j}}} = [\overline{LT}]\left[\frac{[\overline{LV_{i,j}}]}{\sqrt{\lambda_{Li,j}}}\right] \tag{18}$$

The left hand side of Eq. 18 are standard normal variates of the predictor variables $L_{i,j}$ for $i \neq j$ and $i=1, 2, 3$ having zero mean and unit standard deviation. Here $\mu_{\bar{L}_{i,j}}$ and $\sigma_{\bar{L}_{i,j}}$ are the parameters of the lognormal distribution. The correlation coefficient of the standard normal variates is now $\rho_{\overline{L_{1,j}}}$, where $\rho_{\overline{L_{i,j}}}$ = F parameter * $\rho_{L_{i,j}}$[Kiureghian, et al, 1985]. The F-parameter for Eq. 13 in the case of large (n=90) and normally distributed pH data is unity [Haldar et al, 2000].

The general multivariate approach in Eq. 18 for removing the correlation between the predictor variables making them independent is provided in Appendix. A.2.

Predictor Variables	θ	m_1	m_2	m_3	m_4	m_5	R
L_1	1.67	0.824	-0.318	-0.0814	0.405	-0.733	97.9

Table 5.9: Multivariate model constants for Eq.15, $N = \frac{L_1}{\theta+m_1L_1+m_2L_2+m_3L_3+m_4L_4+m_5L_5}$

Table 5.9 provides results of a consolidated study irrespective of the manufacturing of raw material, process, or geospatial property changes inherent in the porous filter vessel. Table 5.9 tabulates the model constants m_1, m_2, m_3, m_4 and m_5 which elaborate the individual influences of the predictor random variables, L_1, L_2, L_3, L_4 and L_5. The model constant m_2 is negative which indicates a negative effect of flow rate L_2 on N (ΔpH). This confirms that diffusion changes with time. Encouraging R^2 value indicates a high degree of linearity in the change in pH as a function of independent variables of L_1, L_2, L_3, L_4 and L_5. The difference in turbidity parameter L_1 has maximum impact on the pH value. A non-parametric analysis of variance validation of the model is shown as:

$$Var(G) \approx (m_1^2)VarL_1 + (m_2^2)VarL_2 + (m_3^2)\,VarL_3 + (m_4^2)\,VarL_4 + (m_5^2)\,VarL_5 \tag{22}$$

$$1.992 \approx 1.4054 + 0.0113 + 0.0147 + 0.0242 + 0.040 \tag{23}$$

It is very clear from Eqs. 22 and 23 that turbidity plays an important role in defining the pH increase in the filtrate of a PCCW filter. Organic and inorganic compounds such as humic acid and flux ions such as aluminium, barium, copper, manganese, silicon, antimony, etc., of the clay filter material affecting the pH. Microbial organics and minute particles of clay are in the form of colloidal particles which are other major contributors to the increase in pH.

5.4 Conclusions

Multivariate stochastic models have been proposed to predict leaching characteristics in PCCW filters manufactured locally in Ghana, Cambodia, and Nicaragua. New models connecting flow with material as well as compositioinal aspects of fluid varying with time are proposed. Three different cases have been studied. Results are summarized as follows:

1. In Case 1, it is found that leaching characteristics, i.e., pH increase is influenced by time of filtration, as well as the type of porous material.
2. In Case II, the non-equilibrium thermodynamics parameters based on the Onasager assumption are simulated with simple measurable parameters of turbidity, temperature, mass flow rate and electrical conductivity.
3. Reciprocity theorems proved and proposed by Onasager are confirmed with the help of multi-parameter correlation coefficients. A new linear stochastic regression hypothesis has been enumerated for the prediction of positive changes in pH during percolation through a PCCW with a specific composition.
4. Case III of this gravity-driven flow study looks into pH change as a function of varied material composition of the PCCWs and time of percolation through the PCCWs.
5. A new dependent variable transformation linear model, has been proposed for predicting increase in the response variable. Influent water turbidity influences the increase in filtrate pH. Flow rate increase decreases the pH of the system.

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Chapter 6: A Field Study on the Use of Porous Clay Ceramic Filters And Influences on the Public Health of Eweje Village

Abstract

A study of the organic material based clay ceramic filters for water purification has been undertaken in a rural setting in Africa. A total of 52 families were distributed filters at Eweje Village, Odeda local government area, Ogun State, Nigeria. A maintenance plan was provided for the use and proper maintenance of the ceramic filters for filtration trails as well as for future usage. Surveys were carried out at Eweje containing socio-economic, educational and behavioral questions influencing the acceptance and use of porous clay ceramic filters. Several parameters studied include time of use of filter, maintainability, education, societal economics, and social the status of the people using the filters. These parameters were carefully selected and surveys were done for the past year.

There was an interdependence of each of these parameters studied in this technical chapter. It was found that health of the Eweje Village community was greatly influenced by the number of people using the filter, the length of filter usage, medical advice or traditional medication knowledge, and economic status in decreasing order of influence.

6.1 Introduction

Eight hundred eighty-four million people are still without potable water with only 5 years to go to meet the Millennium Development Goal of the WHO-UN [WHO, 2010]. It is also very important to note that 34% of the deprived people live in Sub-Saharan Africa. Also in the last 18 years there has been 10% increase in total population of those who have access to potable water [WHO, 2010]. As per predictions by the World Bank in 2003, by 2015, 5-10% of the population of Middle East and North Africa, Latin America and the Caribbean will still be without reliable potable water. Similarly, approximately 15% of South Asia and 25% of Sub Saharan Africa will not have access to potable water resources [Hillie et al, 2009], Several water filtration technologies will be started by educational initiatives and non-governmental organization in the near future to resolve potable water scarcity [Sobsey et al, 2008]. Chlorination with safe storage, chemical coagulants such as WaterMaker (Control Chemical , Alexandria, VA), PuR (Proctor and Gamble, Cincinnati, OH), sodium hypochlorite (SFH/Nigeria) , nut/seed organic medicinal materials, sunlight exposure techniques such as SODIS, SOLAIR, UV radiation techniques, filtration techniques such as nano-membrane filtration, reverse osmosis technique, Pureit (HLL Ltd., Unilever Inc., India), organic additive based ceramic filters, Kanchan MIT arsenic filter and bio-sand filters are some of the most studied and surveyed techniques used around the globe for water purification [CDC , 2008, Clasen et al, 2006, Clasen et al, 2007, Hillie et al, 2009, Olatokun et al, 2009, Ngai et al, 2006, Sobsey et al, 2007, Sobsey et al, 2008].

Education initiatives have been taken in Nigeria near Sub-Saharan Africa under the research auspices of Potters for Peace, Princeton University and Ohio State University. UNICEF joined with a local non-governmental organization in Myanmar formed the Community Development Association to initiate water purification technology to the household level [Naing, 2007]. More than 3,000 ceramic water filters have been distributed in the Phyu village and schools in Myanmar. More than 80% of the households near the delta and coastal areas use these filters regularly and customer satisfaction is about 90% [Naing, 2007]. With this ceramic filter any particle or organisms that are larger than 1 micron are trapped in the filter.

Millions of ceramic filters are in use in several countries in African, Asian, and South American continents. Studies on performance of clay ceramic filters in Bolivia conducted under the nongovernmental organization Food for the Hungry International showed a decrease in the cases of diarrhea by around 45% [Clansen et al, 2006]. In the studies conducted by Sobsey et al in 2008, ceramic filters and biosand filters were found to best fit the sustainability criteria in the field with consumers [Sobsey et al, 2008].

The main objectives of the socioeconomic, educational and behavioral study were,

a) To assess the microbial efficiency of the filters manufactured at Priceton University. Similar filters of high degree of performance are installed in community homes at Eweje Village, Odeda Local Government Area, Abeokuta, Ogun State, Nigeria, and West Africa.
b) To assess the health impact due to use of these ceramic filters.
c) To evaluate which of the economic and social parameters in Eweje influenced the health of the people using ceramic filters for more than 6 months.
d) Study the interaction between the social and economic variables arising in this study which were predictors for health status of people using ceramic filters in Eweje.

6.2 Methods

6.2.1 Manufacturing Procedure

Ceramic water filters were manufactured with locally available clay and sawdust. These filters were low cost but considered as efficient and sustainable technology to treat drinking water in developing countries [Sobsey et al, 2008].

The porous clay ceramic ware (PCCW) filters were manufactured from moistened suspensions containing clay – sawdust (C-S) in 45-55, 65-35, 50-50, 55-45 ratio by volume. Due to the plasticity of the moistened clay-sawdust blend, it could mold under stress to any shape as required. The filters were cast in the shape of a frustum [Donachy, 2004]. Sintering these filter molds to around 900°C introduces numerous pores into the mold serving its filtration capabilities [Lee et al. 2001; Franz, 2005; Dies, 2003; Oyanedel-Craver and Smith, 2008]. The flow characteristics of the 50-50 filter were far better than that from the other C-S ratios mentioned above [Lee et al, 2001]. Hence the PCCW 50 -50 filters were chose for use in the survey. These frustum shaped filters had length of axis dimensions of 26cm, lower base diameters of 20cm, and upper base diameters of 23cm respectively. The filter wall and base had a thickness of 0.5cm and 1cm respectively.

6.2.2 Microbial Removal Efficiency test for the PCCWs

Before distribution of the 50-50 PCCW filters for the field testing and survey, microbial filtration experiments were performed on series of PCCW filters of 45-55, 50-50 55-45, and 65-35 volume ratios. To determine the filtration efficiency, 10–20 ml cultures of the non-pathogenic *Escherichia coli* K-12 strain W3110 were grown in Miller's LB Broth at 37° C for 18–24 hrs with vigorous aeration either by shaking at approximately 200 – 220 rpm or by stirring (VWR digital stirrer/hotplate). This testing was carried out at Mechanical and Aerospace department at Princeton University, NJ, USA.

Four milliliters of this stationary phase culture were mixed into 4 L of sterile purified water, producing a pre-filtrate suspension containing 10^6 to 10^7 cells/ ml. The entire 4 L of pre-filtrate was poured rapidly into a water-saturated filter, and 3–4 L of the filtrate was collected in a 5 gallon plastic pail lined with sterile plastic. The numbers of viable cells in the pre-filtrate and filtrate suspensions were determined by appropriate dilution into sterilized purified water and plating onto Miller's LB agar.

The colonies were counted after overnight incubation at 37°C and used to calculate viable cells/ml. If the viable count of the filtrate was low, cells present in larger filtrate samples (10 - 100 ml) were collected using sterile filtration assemblies (Millipore). The filter was then removed from the filtration assembly, placed directly onto Miller's LB agar and incubated overnight at 37^{o}C. To decontaminate filters between experiments, filters were either rinsed thoroughly with purified water and dried in full sunlight for 5–8 hrs or rinsed with 95% ethanol followed by drying at room temperature.

The efficiency of *E. coli* filtration was calculated as shown below. First, the final *E. coli* concentration was calculated from:

$$\eta_{E.coli} = 1 - \frac{N_f}{N_{pf}}$$

where N_f is the number of viable cells per milli-Liter of filtrate and N_{pf} is the number of viable cells per mililiter in the pre-filtrate. The log reduction value is defined as [Lantagne,
2001],$LRV =$
$log_{10}\,(Viable\ E.coli\ count\ in\ Pre - filtrate/Viable\ E.coli\ count\ in\ filtrate)$.

6.2.3 Setting

For testing these PCCW filters in real life conditions in a developing country these filters were manufactured and tested in Eweje, Odeda Local Government Area, Ward 1, Abeokuta, Ogun State, Nigeria, West Africa [OSG, OdedaLGA, 2010].

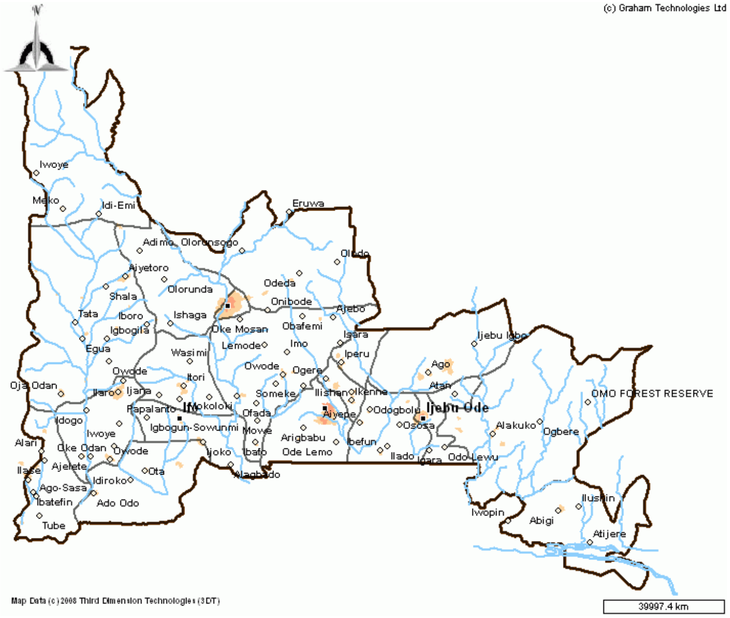

Figure 6.1: The map of Ogun State in Nigeria showing Odeda which is located in the north central region of Ogun State [Courtesy: Ogun Land Information Systems, Ogun State Bureau of Lands and Survey].

Odeda local government shares boundaries with South Abeokuta in the south, North Abeokuta in the west, Obafemi Owode local governments in the east, and Oyo state in the north respectively. The climate is tropical with heavy rainfall from April to July and in September and October. Average temperature is about 32°C but humidity is high at 95% [OSG Odeda LGA, 2010]. It is very important to know the climate before surveying health since climatic change is a very pertinent factor influencing the health of the population.

Collaborators from Princeton University, University of Agriculture, Abeokuta, Ogun State, Nigeria and graduate students went to every family in Eweje for this survey. The main sources of water are ponds and rivulets running near Eweje village.

Manufactured ceramic filters were distributed to 53 families for free in February 2009. These families were surveyed on the effectiveness of the ceramic filters in purifying water. The survey was initiated in February 2009 when the filters were distributed. Survey was carried out every 3 months while the filters were utilized by the people. The survey was quantitative and qualitative in nature with an objective to reveal the effects of the ceramic filter based water treatment on the social and economic status as well as the health of the people surveyed. It was important to note that several people moved out of the Eweje locality to cities due to job and employment opportunities in the course of the 6 months during the survey. All of them took the filter along with them. There was only one person who resisted participating in this survey in the locality who did not believe in any new technology. So the results of 30 families have been documented in this study.

6.2.4 Personnel

Collaborators from Princeton University, University of Agriculture, Abeokuta, Ogun State, Nigeria and graduate students were involved in data collection, PCCW filter use dissemination, and health survey. The Ministry of Health, Nigeria played an important role in the health survey by allocating a resident doctor, Dr. Ogunyale, from the ministry to be a part of this project. He provided important inputs while preparing the survey sheet for the people residing in Eweje Village. The visits of personnel were devoted primarily to educate the people, interview the population of filter users based on questions on the survey sheet. Each of the visits were carried out by a team comprising of two students acting as the investigator and coordinator. For each visit the team filled out duplicate copies of the questionnaire in their native Yuroba tongue as well as in English.

6.2.5 The Questionnaires

It is very important to have a reasonably significant population for such a survey. A reasonable sample set of 53 families were surveyed on the effectiveness of the potable water filter. The survey was discussed and explained individually to the user of the PCCW filter. The consent of the user was obtained. The questionnaire contained questions pertaining to economic status, level of education, family demographics, individual hygiene and health concerns before and after PCCW filter use, availability of medicine or medical consultation facility, potable water source, PCCW filter use and cleaning frequency, and number of PCCW filter units in use per family (Appendix). This also became the major parameters being studied in this technical chapter to influence health.

It is very important to note that health issues may be due to multiple factors so it is assumed to have lognormal characteristics. The survey looked at multiple parameters. Health of the people was predicted with the help of 8 parameters which were expected to closely influence the filter usage. The number of filters for each family X_1, filter cleaning frequency X_2, children below 5 years of age X_3 , members in a family X_4, and time of filter usage X_8, are five of the quantitative predictor variables used in this study. Qualitative predictor variables were wealth statistics $X_{5,}$ educational qualification X_6 and availability of medical consultation X_7. Qualitative variables were expressed in percentages assuming and evaluating their knowledge level, material assets and their approach in dealing with health issues respectively. The criteria for judging these qualitative variables were developed and agreed by the collaborators from Princeton University, Ohio State University, University of Agriculture, Abeokuta, Nigeria and Dr. Ogunyale from the Ministry of Health, Nigerian federal government.

6.3 Results

6.3.1 Microbial Removal Efficiency results for the PCCWs filters tested at Princeton University

The percentage of *E. coli* removed by PCCW filters are presented in Table 6.1 below. Note that the ranges are from duplicate experiments that were performed on the same specimens. All of the filters exhibited very high *E. coli* removal rates. In comparison, the filtration efficiency of a typical 50-50 colloidal silver coated filter produced by Potters for Peace was 99.99%, the 50-50 filter and 65-35 filters without colloidal silver coating manufactured at Princeton University had very close filtration efficiencies of 99.99 ± 0.00 and 99.97 ± 0.03% respectively [Lantagne, 2001, Bielefeldt, et al, 2009]]. This is enumerated in Table 6.1 below.

PCCW Filter	Test 1 $\eta_{1E.coli}\%$	Test 2 $\eta_{2E.coli}\%$	Average Efficiency ± Range(%)	Log Reduction Value-Test 1	Log Reduction Value-Test 2
45:55	99.97	99.85	99.91 ± 0.06	3.49	2.84
50:50	99.99	99.93	99.97 ± 0.03	8.16	3.15
55:45	99.52	99.84	99.68 ± 0.16	2.314	2.79
65:35	99.99	99.99	99.99 ± 0.00	5	6.9

Table 6.1: Bacterial filtration results obtained for filters with different clay to saw dust ratios.

It was earlier mentioned that 50-50 filter was having a comparatively better flow characteristic as compared to other PCCW filters. And from table 6.1 it is found that in test 1, 50-50 filters had competitive microbial filtration efficiency and was better than EPA standard LRV value of 6 for bacterial removal [Clansen et al, 2009]. Oyanedel-Craver et al, 2008 and Bielefeldt et al, 2009 reported bacterial removal greater than >2.5 log and > 3 of *E. coli* pulse-spiked onto 50-50 ceramic ware respectively which is very much comparable with the results in Table 6.1. Similarly Lantagne (2001) reported 98.2, 97.0, and 82% removal of total coliform, fecal coliform, and fecal streptococcus, respectively, by using filters from Potters For Peace, Nicaragua. It should be noted that there is no silver coating on the filters. Hence 50-50 filters are estimated to provide better service in the field also.

6.3.2 Survey results:

Population is a major parameter without which the survey of the effectiveness test of the PCCW filters cannot be carried out. It is found that 30 people responded to the survey regularly for more than 6 months. The other 23 people out of the 53 person sample population under survey were able to provide their inputs only once at the initiation of the survey. Hence their comments have been neglected in the studies. The health status of the 30 families were recorded in percentage and plotted in Fig.6.2.

From the Fig. 6.2, it is clear that that with increase in the number of people in a family P, there was better health. It was estimated by World Health Organization in 2002, that 9/10 deaths were children and 54.2 million disability adjusted life years were lost due to unhygienic condition and scarcity of potable water [Parikh et al, 1999, WHO, 2002].

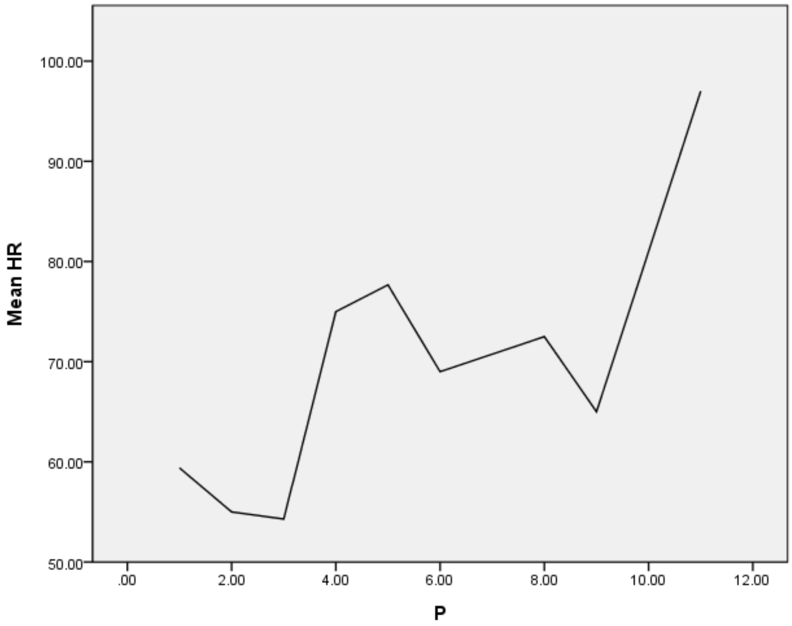

Figure 6.2: The variation of health (HR %) as a function of the size of family (P) 6 months after initiation of PCCW water filter use in Eweje Village, near Lagos, Nigeria.

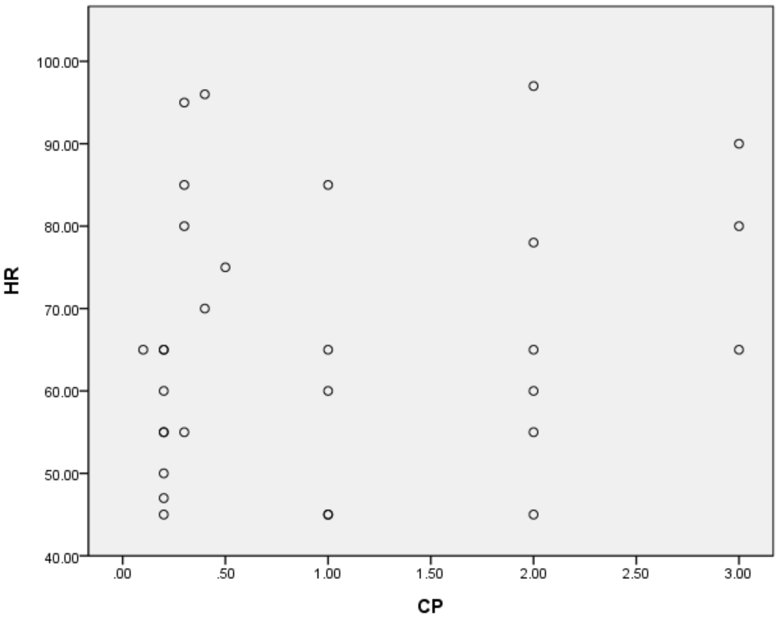

Figure 6.3: The variation of health (HR in %) as a function of number of children below 5 years of age (CP = X_3).

In Fig.6.3, the X axis represents the distribution of the number of children below 5 years of age. It was found that children below 5 year of age (CP) helped in making the health response (HR) a very random variable. It is important to note that presence of a child within the family made a large difference in the health status. It should be noted that children and mothers were more susceptible to health problems compared to others in the family residing in rural areas in developing nations [Montgomery et al, 2007]. It is also true that women and children spent much time fetching potable water preventing them from attending schools [Jalan, et al, 2003 and Hillie et al, 2009].

There is a significant influence of wealth on a family decision making either to consult a doctor or to adopt some type of purified drinking water for them, such as bottled mineral water. It is observed that even when family income was high there was no predictable improvement in health. This is very clear from Fig 6.4, which supports this statement.

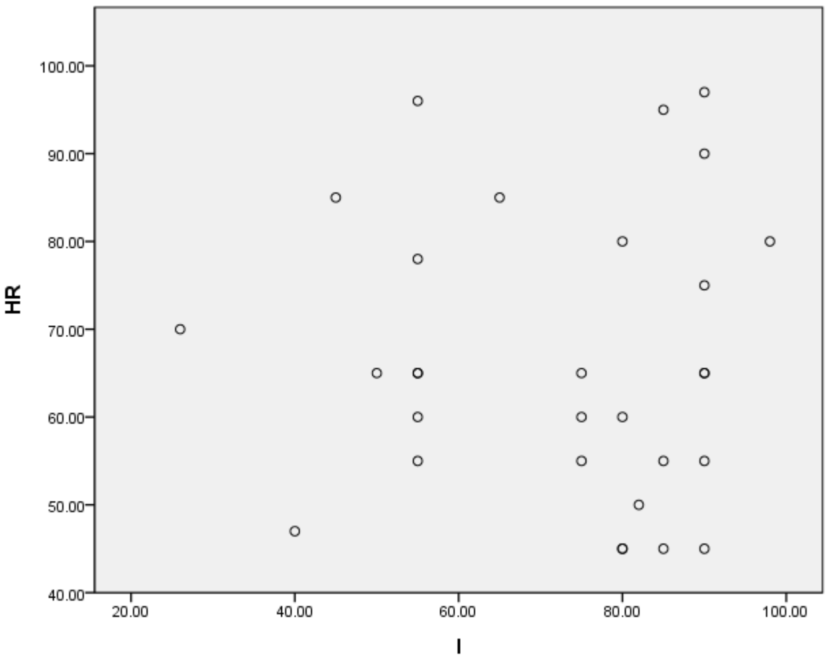

Figure 6.4: The variation of health response (HR) as a function of family income ($I = X_5$) expressed in percentage

The families with higher economic status were found to either have sound or worse health characteristics. This may be due to other influencing parameters such as presence of children below 5 years of age or low maintainabilityof filter, M,. The other factor which may mainly contribute to affect health is education. It has been noted that education of females in the family proved to be a positive influence to heath of the family [Jalan et al, 2003].

It was found from the survey that with increase in educational qualifications the awareness of the people to clean the filter was higher. This awareness as plotted in Fig. 6.5, leads to better condition of health arising from eradication of water borne diseases owing to use of the new filters.

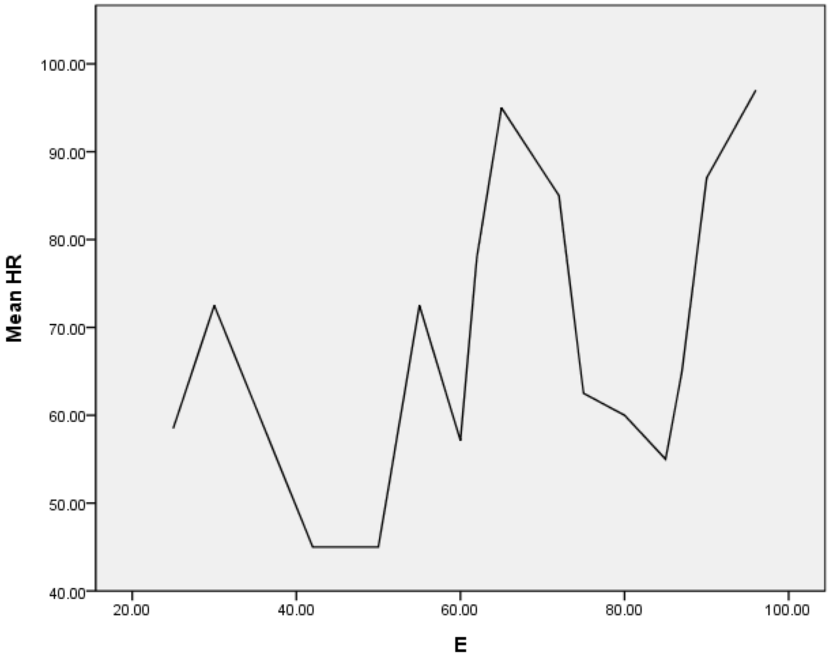

Figure 6.5: The variation of health (HR) as a function of Family Education (E).

It is found that health of families with people having 20-60% level of education (less than or equal to third standard in school) contributed to low health percentage. People with more than 60% level of education (above fourth grade to degree certificate) did have better health.

It is found that loosing school hours was deleterious to health [UNICEF, 2007]. Education is the only edge to restore a sense of normalcy when illiteracy influences disastrous health. For that reason the WASH programs in schools in countries like Thailand, Mongolia and Niger which imparted awareness to hand washing with soap and other sanitation methodology to its students [UNICEF 2008, UNICEF 2009]. Lack of potable water and sanitation facilities in schools prevented female attendance and impacted their learning environment [Hillie et al, 2009, UNICEF, 2009]. Awareness is considered a way to make people realize the importance of better hygiene practices like hand washing and should be a part of the curriculum in schools throughout developing countries [MNN, 2010]. We can find that an awareness of the importance of pure drinking water and the exposure to mass media has significantly affected people in developing countries. As a result, the number of people who died of cholera has decreased from 8,500 in 2007 to 42 in 2009 in Africa [MNN, 2010].

Hence the awareness to hygiene will really improve usage and maintainability, M, of the PCCW filters. From Fig. 6.6, it is seen that with increase in maintainability, M (Filter cleaning frequency X_2), health of the people was seen to improve proportionally.

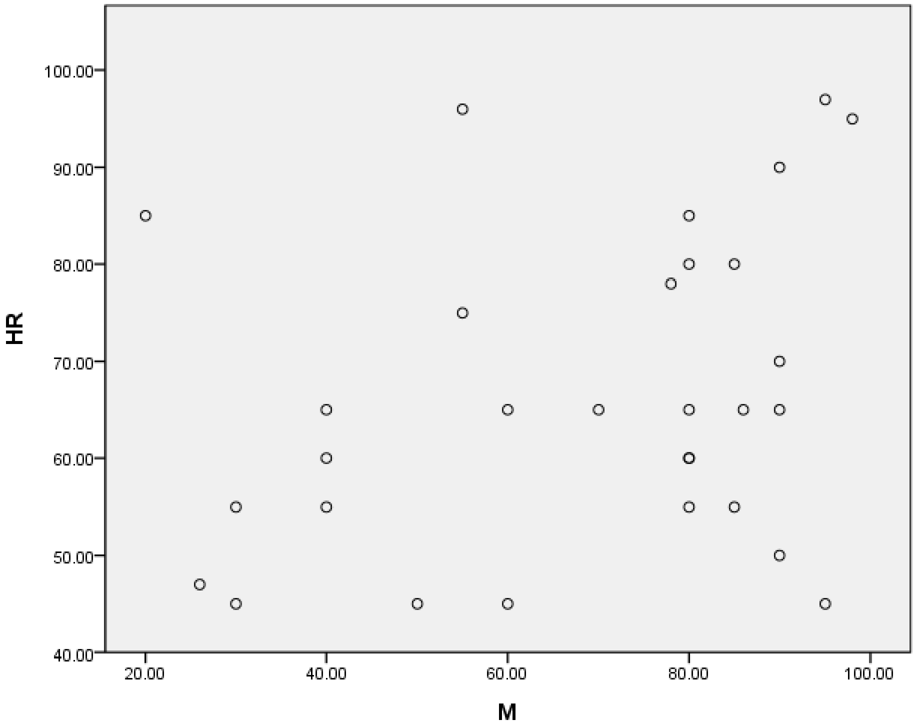

Figure 6.6: The variation of health response (HR) as a function of PCCW water filter maintenance M.

6.4 Theoretical Development

A non linear behavior of Health Response ($HR = Y$) is seen from the above results from Fig 6.2 to Fig 6.6. A lognormal stochastic multi-parameter model has been proposed in this research to model the health response, Y, of the people at Eweje Village, Lagos State, Nigeria.

The parameters X_1, X_2, ….. X_k are manifestations of societal, population, technological, and economic infrastructure influencing the health changes in Eweje due to filter use. So, health response, Y, can be expressed mathematically as follows,

$$Y_i/Y_{i-1} = X_i^{b_i} \text{ for } i = 1, 2,3\ldots..k \qquad (1)$$

The above expression of X_i^{bi} is known as the transfer function. This function mathematically expresses the step-by-step effects of the variables mentioned above with time on the health of the community in Eweje [Soboyejo et al, 2000]. This nonlinear behavior is simulated with a stochastic step function from step 0 to step 1 for one of the predictor variables as shown below in Eq. 2.

$$Y_1/Y_0 = X_1^{b_1} \text{ for } i = 1, 2,3 \qquad (2)$$

where $Y_0 = a$ initial value of the stochastic process model, and which is a model constant in this problem. The Eq. 2 is extended by increasing the number of parameters and is written in a general form,

$$\frac{Y_n}{Y_{n-1}} = X_n^{b_n} \qquad (3)$$

$$Y = Y_n = Y_{n-1} X_n^{b_n} = a X_1^{b_1} X_2^{b_2} \ldots\ldots. X_n^{n} = a \prod_{i=1}^{n=k} X_i^{b_i} \qquad \text{For n = 1, 2,…..k.} \qquad (4)$$

Since the predictor random variables have different dimensions, Eq. 4 can be mathematically reformulated as

$$Y = Y_o \prod_{i-1}^{k} (X_i/X_{io})^{b_i} \qquad (5)$$

$$a = Y_o\left\{\prod_{i=1}^{k} X_i^{b_i}\right\}^{-1} \quad (6)$$

where X_{io} is any reference constant with the same units as X_i.

Eq. 4, can be expressed as

$$y_i = \ln Y_i = \ln a + \sum_{i=1}^{k} b_i \ln X_i \quad (7)$$

From Table 6.2, it is found that X_4, number of members in the family, affects positively the health, Y, due to high frequency in cleaning the filters in highly populated families. This is very clearly enumerated in Fig 6.7.

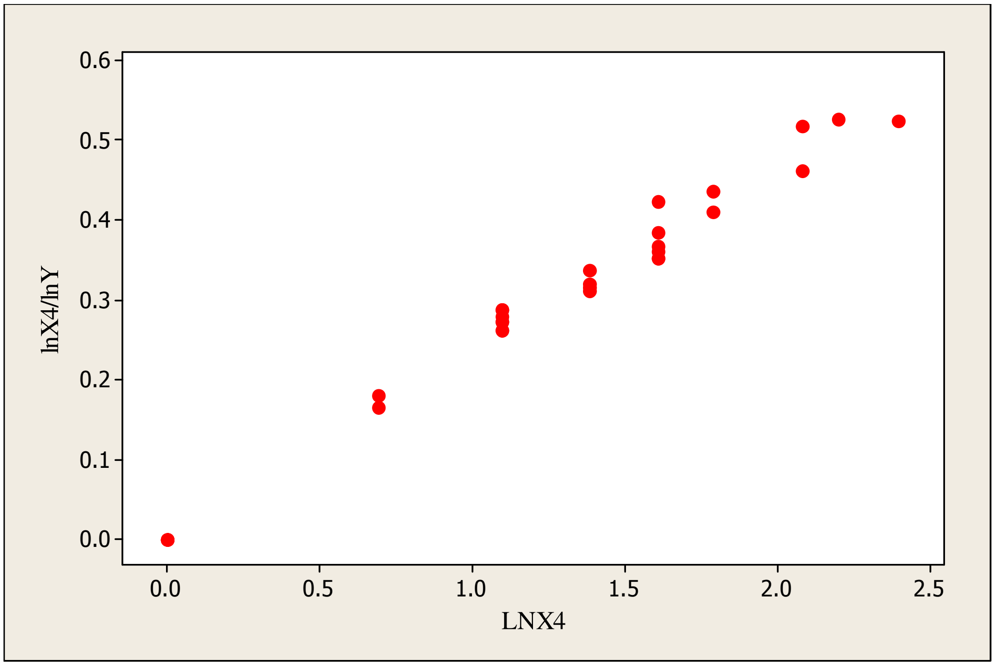

Figure 6.7: Scatter Plot of the new transformed variable X_4/Y as a function of Y.

Hence a predictor variable transformation in the response variable is done. Then the equation can be written as

$$G = X_4/Y = aX_1^{b_1}X_2^{b_2} \ldots\ldots\ldots . X_8^{b_8} \qquad (8)$$

Here the predictor variable transformation is applied such that it does not affect the actual behavior of the response variable Y.

To test the validity of the assumed transformation X_4/Y over multiplicative transformation of Y as well as raw data Y, a Kolmogorov Smirnov goodness of fit test was performed at a 99% level of confidence and is shown in Table 6.2 below.

Kolmogorov Smirnov Test at α=0.01					Critical ValueD_n^α
	Y	Ln Y	Ln G	n	
D_n	0.173	0.127	0.2	30	0.29
p value	0.03	0.15	0.009	30	0.01

Table 6.2: D_n and D_n^α for Kolmogorov Smirnov Test for the different models for the Health Response Y [Ang et al, 1975].

Here if the D_n value is less than the critical D_n^α at α=0.01, the proposed distribution for the variable is accepted [Ang et al, 1975].

All the variable predictor parameters for health response (HR=Y) were highly correlated to each other and has been enumerated in Table 6.3 below. It is found that filter cleaning frequency X_2 is highly correlated with number of members per family X_4. It is clear from the correlation Table 6.3, that educational awareness X_5 is found to increase with increase in wealth X_6 and vice versa. This is because better education provided people with high paying jobs, or people with enough money were able to avail good education.

	$\rho1$	$\rho2$	$\rho3$	$\rho4$	$\rho5$	$\rho6$	$\rho7$
$\rho2$	0.23						
$\rho3$	0.04	0.406					
$\rho4$	0.149	0.508	0.734				
$\rho5$	-0.032	0.421	0.339	0.482			
$\rho6$	-0.015	0.16	0.123	0.568	0.55		
$\rho7$	0.01	0.03	-0.263	0.02	0.064	0.441	
$\rho8$	0.065	0.41	0.208	0.313	0.288	0.181	0.035

Table 6.3: Correlation coefficients ρ_{ij} for each pair of the random parameters influencing health namely, the number of filters for each family X_1, filter cleaning frequency X_2, children below 5 years of age X_3, members in a family X_4, wealth statistics X_5, educational qualification X_6, and availability of medical consultation X_7 and time of filter usage X_8.

The correlated predictor matrix column elements in Table 6.3, with correlation coefficient $\rho_{X_{i,j}}$are linearly transformed to the independent variables V_i and are scaled as [Haldhar et al, 2000, Krishnamoorthy, 2006].

$$\frac{\left(\left[\overline{lnX}_{i,j}-\mu_{\overline{X_{\iota,J}}}\right]\right)}{\zeta_{\overline{X_{\iota,J}}}} = [\bar{T}]\left[\frac{\left[\overline{V_{\iota,J}}\right]}{\sqrt{\lambda_{i,j}}}\right] \tag{9}$$

The left hand side of Eq. 9, are standard normal variates of the predictor variables $\ln X_{i,j}$ for $i \neq j$ and $i=1, 2, 3$ having zero mean and unit standard deviation. Here $\mu_{\overline{X_{\iota,J}}}$ and $\sigma_{\overline{X_{\iota,J}}}$ are the parameters of lognormal distribution.

$[\bar{T}] = [\emptyset]^t$, which constitutes all the principal components in the transformation matrix $[\bar{T}]$. The $V_{i,j}$ for i=1,2,3 in Eq. 9, are the independent normal predictor variables with $\lambda_{i,j}$ for i=1, 2, 3 the corresponding variances. An explanation of the procedure in Eq 9 is elaborated in the Appendix.

6.5 Results: Influences of the predictor variables on health status of people at Eweje

The multiplicative nature of Y is preserved and the multivariate approach developed above is applied to fulfill the assumptions of independent variables for regression.

The multivariate approach helps in identifying the actual independent influences of the predictor variables on the newly transformed response variable G which now consolidates the health parameter Y. So the regression model coefficients are shown in Table 6.4 below.

Model	$\bar{a}$	$\overline{b_1}$	$\overline{b_2}$	$\overline{b_3}$	$\overline{b_4}$	$\overline{b_5}$	$\overline{b_6}$	$\overline{b_7}$	$\overline{b_8}$	R^2	S
G	0.29	0.0842	-0.018	-0.022	-0.027	-0.024	-0.014	-0.030	-0.103	99	0.0184

Table 6.4: Multivariate model constants for Eq. 8 , $G = X_4/Y = aX_1^{b_1}X_2^{b_2} \dots \dots \dots . X_8^{b_8}$.

From Table 6.4, it is clear that model constants which are corresponding coefficients of the predictor variables in the Eq. 13 vary as,

$$\overline{b_1} > \overline{b_6} > \overline{b_2} > \overline{b_3} > \overline{b_5} > \overline{b_4} > \overline{b_7} > \overline{b_8}$$

This would mean that apart from population X_4 which is a major influencing parameter, the other highly influencing parameters can be written as below in their increasing order of influence.

$X_1 < X_6 < X_2 < X_3 < X_5 < X_7 < X_8$

It is found that with time for which PCCW filter is used X_8, there has been a steady decline in health issues in the Eweje locality. This confirms a long term effect of better potable water from the PCCW filters on health [Brown et al, 2007]. This also predicts the usage of the filters with satisfaction. Secondly, people who had close access to medical facilities X_7 prevented themselves from diseases. This means that people did see their local medical officer for his/her advice on health issues. It is very important to note that even though Odeda people are less educated academically they have good knowledge of traditional medicine [Olatokun et al, 2009]. Mostly women have this knowledge base who happen to be the primary water collectors [Olatokun et al, 2009, McCarton, 2009].

Financial status X_5 as well as money did have a prominent effect in determining the health trends in Eweje but was less influential than availability of medical facilities and duration for which the filters were used. It is very important to find X_5 as the third major influence confirming the finding by WHO that 1.8 million water related diseases were basically from low income countries. This actually provides an insight on basic psychology of the people swaying away from buying expensive new technology to avail potable water.

Education and awareness played a pertinent role but not to expectation [Jalan et al, 2003]. This would support a requirement for vigorous awareness programs educating people about sanitation, water and hygiene and their correlations [UNICEF, 2010].

This novel approach has been used for identifying the multiple parameters such as monetary status, knowledge level, and consciousness towards better medical treatment, time of usage of the filter or other devices, demographics and inclination of rural people to adapt new technology, which are found to affect the health of the society. This is an important ongoing research project. The public health impact of this project will have beneficial practical application in water quality improvement in rural areas of the world.

6.6 Conclusion

Multivariate stochastic regression formulation was used for identifying the individual socio-economic parameters affecting health of the society at Eweje Village, Ogun State, Nigeria. Summarizing the major outcomes of the health response effects due to clay ceramic water filter use as explained below.

1. The duration of filter usage has been a major parameter for influencing general health at Eweje Village.
2. Accessibility of medical facilities or knowledge of traditional medicine within the people at Eweje had a great influence on the health outcome due to filter usage. The knowledge of traditional medicine should be accounted under the parameter education.
3. Financial status played a major role in influencing health at Eweje.
4. Random parameters influenced health in an order as shown below,

   Number of filters for each family, X_1 < educational qualification, X_6 < filter cleaning frequency, X_2 < children below 5 years of age, X_3 < wealth statistics, X_5 < availability of medical consultation, X_7 < time of filter usage $X_{8,}$ < members in a family X_4.

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Chapter 7: An Overall Conclusion and Future Work

This document documents a part of a large ongoing project on technology optimization for providing a viable low cost feasible technology which could withstand the challenge of future pollutants and associated looming scarcity of potable water.
A brief overview of sustainable clay ceramic water filter has been provided in Chapter 2. It provides the review on the different clay ceramic filter (CCF), raw materials of manufacture and respective research references. The variation in flow with time in the CCF was studied and stochastically assessed in Chapter 3. A lognormal model with a predictor transformation of response was found capable for predicting the cumulative discharge from the CCF with variant raw material quantities. Similarly model with a predictor transformation of response was found capable for predicting the cumulative discharge from the CCF as a function of time.

The strength of ceramic porous media has been studied for monotonic tensile, flexural and compressive loading events. This was done to assess the behavior of clay matrix when as organic additive is added to it. A lognormal model has been derived for predicting the strength of clay ceramics. Micro-structural properties were confirmed to have maximum influence on the tensile mechanical properties of ceramics.

The material flows that may occur during the forming process of the clay ceramic were found to improve the strength of clay ceramics. This indicated a crack shielding phenomenon occurring with additive alignment during material flow while manufacturing. Materials formed through this phenomenon did not have a large influence of micro-structural geometry on their strengths. Compressive strength of clay ceramic was found to decrease linearly with increase ceramic composite porosity.

Leaching characteristics in clay ceramics are influenced by time of filtration process as well as the type of porous material. Chapter 5 also confirms the reciprocity theorems proved and proposed by Onasager with the help of multi-parameter correlation coefficients.

A new linear stochastic regression hypothesis has been enumerated for the prediction of positive change in pH during percolation through a CCF with a specific composition. Gravity driven flow study through CCF looks into pH change as a function of thermodynamic parameters and time of percolation through the PCCWs. Chapter 5 also developed a new dependent variable transformation linear model for predicting increase in the alkalinity.

Chapter 6 confirmed the efficacy and utilization of the filter in reduction of water based diseases on field at Eweje Village, Ogun State, Nigeria. Filter use, knowledge or medical accessibility and financial status influenced the health response at Eweje.

7.1 Future Work

New materials are to be tested for removing future pollutant in water. Filters have to be made structurally rigid incorporating material that better glue clay and organic material together and prevent breaking during transport. While working on this it is required to provide with a material which might also contribute to purification of water. Similarly additives must be able to prevent leaching or wasting og water filter and causing internal water contamination.

Microbial efficacies of the filter need to be improved to handle more potent microbes with superior survival features.

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Appendix

Part 1

Assuming the cumulative discharge from a filter, Y at any time t, where t>0 and Y_i is a function of random variable X_i; i = 1, 2,….n with an initial value Y_0 at time t = 0. Y_i, the cumulative discharge from a particular porous media filter, is obtained from the step–by-step contributions of n random variables as is also clear from Fig. 4. The cumulative discharge through a given filter is a stochastic birth process, with a well defined asymptotic value Y and is given as [Soboyejo (1965)]

$$Y = \frac{X}{a+bX}$$

Y_i is given by Eq.6 as shown below

$$Y_i = \frac{X_i}{a_i + b_i X_i}$$ for i = 1,2…..n.

The new response variable considered is given by

$$G_i = \frac{X_i}{Y_i}$$

Considering the step–by- step contributions of n random variables it can be shown that

$$G_i = \frac{X_i}{Y_i} = G_{i-1} + b_i X_i$$, then for i = 1 we have

$$G_1 = G_o + b_1 X_1$$

When i = 2

$$G_2 = G_1 + b_2 X_2$$

$$G_2 = G_0 + b_1 X_1 + b_2 X_2$$

.

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$G_n = G_0 + b_1 X_1 + b_2 X_2 + \ldots\ldots + b_n X_n$

Here G_0 = Initial value of G_i , not related to any of the predictors, namely X_i for i = 1, 2, …..n.

G_0 = a, is only related to the material structural properties [Soboyejo (1965)]. And the coefficient of the predictor variables X_i for i = 1, 2, 3…..n are b_1, b_2,……b_n which provides us information about the importance of the influence of each of the predictor variables on response G_i in this case [Bulmer (1979), Soboyejo (1965), Soboyejo (2006)]. These coefficients also represent the variability of G for a unit change in X_i for i = 1, 2, 3…..n [Haldar and Mahadevan (2000)]. It should be noted that with the increase in the number of predictor variables, there is decrease in the deviation or error of prediction by the model as is also seen from the S values in Tab. 2 in this chapter [Halhar and Mahadevan (2000)].

Part 2

From Eq. 13, deviation of predictor variable measurement $lnX_{i,j}$ from the sample mean of each of the predictor variables is written as

$$D = \left[\bar{X}_{i,j} - \mu_{\overline{X_{i,j}}}\right] = \bar{X}_{i,j} - \left(\frac{1}{n} A A^t \bar{X}_{i,j}\right)$$

where, A is auxiliary n x 1 matrix and n is the number of total measurements for each of the k predictor variables. Here$A^t A = \sum_{i=1}^{n} x_i^2 = n$. Then D is written as

$$D = \begin{bmatrix} x_{11} - \overline{x_1} & \cdots & x_{1k} - \overline{x_1} \\ \vdots & \ddots & \vdots \\ x_{n1} - \overline{x_1} & \cdots & x_{nk} - \overline{x_n} \end{bmatrix}$$

The covariance of the predictor variables can be calculated by using the fact that Euclidean Inner product can be written as matrix multiplication (For example, $A.B = A^t B$ where A and B are column vectors) as shown below

$$\left[C_{X_{i,j}}\right] = \frac{1}{n-1} D^T D = \begin{bmatrix} \sum_i (x_{i1} - \overline{x_1})^2 & \cdots & \sum_i (x_{i1} - \overline{x_1})(x_{ik} - \overline{x_k}) \\ \vdots & \ddots & \vdots \\ \sum_i (x_{in} - \overline{x_m})(x_{i1} - \overline{x_1}) & \cdots & \sum_i (x_{im} - \overline{x_m})^2 \end{bmatrix}$$

It is known that correlation values can be derived from covariance values of the predictor variables as depicted below,

$$\rho_{X_{i,j}} = \left[C_{X_i X_j}\right] / [\sigma_{X_i}] \, [\sigma_{X_i}] \tag{17}$$

This can also be written as

$$[\rho_{X_{i,j}}] = \left[C_{X_i X_j}\right] [S_{X_i}][S_{X_i}]$$

$$= \left[C_{X_{i,j}}\right] \begin{bmatrix} 1\Big/\sqrt{\sum_i (x_{i1} - \overline{x_1})^2} & \cdots & 0 \\ \vdots & \ddots & \vdots \\ 0 & \cdots & 1\Big/\sqrt{\sum_i (x_{im} - \overline{x_m})^2} \end{bmatrix} \begin{bmatrix} 1\Big/\sqrt{\sum_i (x_{i1} - \overline{x_1})^2} & \cdots & 0 \\ \vdots & \ddots & \vdots \\ 0 & \cdots & 1\Big/\sqrt{\sum_i (x_{im} - \overline{x_m})^2} \end{bmatrix}$$

Here that $[\rho_{X_{i,j}}]$ is a square matrix, k x k, and if $\left[\theta_{i,j}\right]$ is a non-zero vector in the null space of $[\rho_{X_{i,j}}]$ and exist in the n dimensional space of the predictor variable $X_{i,j}$ data set, then

$$[\rho_{X_{i,j}}] \left[\theta_{i,j}\right] = \lambda_{i,j}\left[\theta_{i,j}\right] = \lambda_{i,j}[I]\left[\theta_{i,j}\right] \tag{19}$$

$$\left[\lambda_{i,j}[I] - [\rho_{X_{i,j}}]\right] \left[\theta_{i,j}\right] = 0 \tag{20}$$

Here $\left[\theta_{i,j}\right]$ is the null space of the characteristic equation term and left hand also represents two orthogonal vectors in the inner product space. It should be noted that $\left[\theta_{i,j}\right]$ contains all vectors perpendicular to the column spaces containing all the predictor variables.

There will be one eigen space$\left[\theta_{i,j}\right]$ for each distinct eigenvalue $\lambda_{i,j}$ for i=1,2..n and j =1,2…k when n>k and so there will be k eigen spaces for $[\rho_{X_{i,j}}]$. Trace of the eigen value matrix also defines the total variance of the predictor variables.

$[\bar{T}]$ is an orthogonal square matrix of size 3x3 on the right hand side of Eq. 13, with normalized eigen vectors $\left[\theta_{i,j}\right]$ for the three participating predictor variables. The normalized eigen vectors can be represented as

$$[\emptyset] = \begin{bmatrix} \left(\theta_1^i\right)^2 / \left(\left(\theta_1^i\right)^2 + \cdots \ldots \ldots \ldots + \left(\theta_k^i\right)^2\right) \\ \vdots \\ \left(\theta_k^i\right)^2 / \left(\left(\theta_1^i\right)^2 + \cdots \ldots \ldots \ldots + \left(\theta_k^i\right)^2\right) \end{bmatrix}^{1/2}$$

Then $[\bar{T}] = [\emptyset]^t$, which constitutes all the principal components in the transformation matrix $[\bar{T}]$. The $V_{i,j}$ for i=1,2,3 in Eq. 13, are the independent normal predictor variables with $\lambda_{i,j}$ for i=1, 2, 3 the corresponding variances.