



ADDIS ABABA UNIVERSITY
Addis Ababa Institute of Technology
School of Chemical and Bio Engineering

**Determination and optimization of Silver Nanoparticle Impregnated
Porous Ceramic water filter for Point of Use Water Treatment**

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This is to certify that the thesis prepared by *Getachew Amogne Ayele*, entitled: Determination and optimization of Silver Nanoparticle Impregnated Porous Ceramic water filter for Point of Use Water Treatment and submitted in partial fulfilment of the requirement for the Degree of Master of Science (Chemical and Bio Engineering) complies with the regulations of the University and meets the accepted standards with respect to originality and quality.

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DECLARATION

I declare that this thesis entitled “Determination and optimization of Silver Nanoparticle Impregnated Porous Ceramic water filter for Point of Use Water Treatment ” has not been submitted in any form for another degree, diploma or an award at any university or other institution of the tertiary education. Whenever contributions of others are involved, every effort is made to indicate this clearly, with due reference to the literature and discussions. Information taken from published and unpublished work of others has been acknowledged in the text and a list of references is given. The work was under the guidance of Dr. Solomon K. (Assistant Professor) instructor in Addis Ababa University, School of Chemical and Bio Engineering.

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Abstract

Clean water is a necessity for healthy human beings so is its provision and access. The porous ceramic water filtration pot has been greatly improving the most contaminated tap water for drinking water in developing countries. This research was carried out to determine the efficiency of the AgNP impregnated porous ceramic water filter pots in improving water from bacterial coliforms. The study was undertaken to evaluate the performance of AgNP impregnated ceramic water filter pots in reducing the E. coli in contaminated water. The effect of firing temperature and volume porosity (P) of a pot-type ceramic water filter on the filtration and E. coli removal efficiency is presented.

The raw materials used to make the porous ceramic pot are clay soil, sawdust and silver nitrate. Clay soil and sawdust were grinded and sieved with 1mm (clay soil), 0.2mm, 0.4mm, 0.6mm and 0.8mm (Sawdust) opening mesh size and then mixed with water and molded in a pot shape and fired at 900 °C, 1000 °C and 1100 °C. The porosity of the ceramic water filters was varied by changing the screen size of sawdust and percentage of sawdust added in the red clay and measured by the absorption test method using Archimedes' principle and the E. coli were tested using the membrane filtration procedure.

The porosity of the filters was found to be directly proportional to the screen size and percentage of the sawdust and slightly inversely proportional to firing temperatures. The filtration rate of water increased with the increase in the porosity of the AgNP impregnated ceramic water filters. Ceramic water filters designs (clay to sawdust mass ratio of 85:15, 90:10, and 95:5) fired at a temperature of 900 °C, 1000 °C, and 1100 °C for eight hours had total E. coli removal efficiency of 85.6 to 99.999% respectively.

Changing in the filter's design or raw materials and the firing temperature will affect the performance of the produced ceramic water filters pots. The results of this study suggest that the mean flow rate for a properly functioning filter (8.1% by mass sawdust and screen size in between 400µm and 600µm) fired at a temperature of 900 °C is 1.652 L/hr. This flow rate is more than the average liters per day for adults as recommended by World Health Organization (WHO). This filter also removed more than 99.96% of E. coli.

Key words: E. coli, silver nanoparticle (AgNP), clay soil, sawdust

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List of acronyms

AgNP	Silver nanoparticles
ANOVA	Analysis of Variance
BHD	Box-Behnken Design
bp	Base pairs
BSF	Bio-Sand Water Filter
CPF	Ceramic pot filter
CSA	Central statistics agency
CWFPs	Ceramic water filter pots
DBPs	Disinfection by products
E. coli	Escherichia coli
HWTS	Household water treatment and safe storage
NGOs	Non-Governmental Organizations
NTU	Nephelometric Turbidity Unit
PAC	Percentage of ash content
POU	Point-of-use
PVP	poly vinylpyrrolidone
SDS	Sodium dodecyl sulphate
SODIS	Solar disinfection
TC	Total coliforms
TTC	Thermo-tolerant coliforms
UNICEF	United Nations International Children's Emergency Fund
UNISDR	United Nations International Strategy for Disaster Reduction
UPV	Ultrasonic pulse velocity
VIF	Variance inflation factor
WHO	World Health Organization

CHAPTER ONE

1. INTRODUCTION

1.1. Background

Water is a basic need essential for the sustenance of all life forms. However, there is a great concern due to the lack of safe and clean drinking water, especially for people living in rural areas of developing countries like Ethiopia. Statistical data's show that worldwide, above 780 million people live without access to safe drinking water and an estimated 2.5 billion number of people lack basic sanitation (Lantagne et al., 2006). Furthermore, yearly approximately 1.8 million people lost their lives due to diarrheal diseases which 90% of these being children under the age of five years, mostly in developing nations found in sub-Saharan Africa, South-East Asia and at some Latin American countries (Dillingham and Guerrant, 2004).

The water crisis is further worsened by the increasing demand for potable water due to increase in population, and pollution of water sources making the water unsuitable for consumption without further treatment (Savage and Diallo, 2005). In spite of these challenges, it is known that improved water supply remarkably reduces death due to diarrhea by 6 – 25 % while improved sanitation reduces it by 32 % (*UNICEF Water, Sanitation and Hygiene Annual Report, 2006*).

The present unique challenges at rural areas in developing countries including Ethiopia are in terms of potable water provision. These include rugged or uneven topographies, the populations distributed over very wide areas, lack of skills for operation and maintenance of water treatment systems, poorly developed infrastructure, and the lack of finances (Onda et al., 2012; Yilma et al., 2009). People living in these areas have to fetch water that is normally untreated from local rivers or dams for use in cooking, washing and other domestic uses. Thus, the provision of safe water using conventional piped water systems to households may be a long way off for such people of the developing world. This means that many of the poorest people must continue collecting raw water and be responsible for treating and storing it at the household level (Joe Brown and Sobsey, 2006). This water is often prone to contamination resulting in outbreak of diseases. It is in response to the problems associated with the resulting waterborne diseases that new strategies for safe water provision in rural areas are being explored. These methods include treating drinking water at the

household level and the devices that can be used are referred to as point-of-use (POU) technologies (Mpenyana-monyatsi et al., 2017; Mwabi et al., 2015). The most commonly used small scale and POU water treatment methods include: boiling; UV disinfection; ozonation; chemical disinfection using chlorine and its derivatives; filtration using ceramic filters; and solar disinfection. However, these technologies have significant drawbacks. For instance, UV disinfection and ozonation require high costs for equipment and energy in order to implement. The use of chemical disinfectants such as chlorine, and iodine leads to the formation of disinfection by products (DBPs) which are carcinogenic and therefore harmful to consumers. In addition, boiling and solar disinfection are time consuming and can only treat in small quantities of water at a time. These factors make the application of these systems in rural areas of developing economies difficult. A relatively simple technology that can potentially overcome these challenges is the use of silver nanoparticle (AgNP) impregnated porous ceramic water filtration and disinfection method.

In Ethiopia at least over half of population still have no access to treated potable water within reasonable distances from their dwellings. Many thousands more take water from open water sources and use it untreated because of problems experienced with provision of adequate and reliable potable water supplies. Further, almost all cities and rural water treatment plants found in Ethiopia are often questioned regarding their design, operation and qualities of water produced. Surface waters have also steadily become more polluted, especially with regard to microbiological quality, which exacerbates the situation of immunocompromised individuals when drinking inadequately treated or poor-quality water. It is an unfortunate fact that the populations still without potable water supply are also the most difficult, expensive and time consuming to service. This leads to people drinking and using water directly from unsuitable water sources, which results in incidences of sickness and even fatalities among young children (CSA, MOWIE, World Bank, UNICEF, 2017).

According to the central statistics agency (CSA) and world bank report in 2017, 35% of the population didn't get improved or safe water for drinking in Ethiopia (CSA, MOWIE, World Bank, UNICEF, 2017). From 60 to 80 percent of communicable diseases are attributed to limited access to safe water and inadequate sanitation and hygiene services. Under five mortality death in Ethiopia is caused by Diarrhea which is the leading cause accounting for 23 percent of all under-five deaths – more than 70,000 children a year (Wash, 2018).

In Ethiopia, there are different types of point-of-use water treatment technologies being used of which boiling, adding bleach, filtration (using porous clothes), and solar disinfection are listed as appropriate point-of-use water treatment methods (Geremew et al., 2018). There has been an effort to improve the coverage of these water treatment options in the country. The Health Transformation Plan of the country (2016–2020) shows that it is targeted to reach 35% coverage in the household use of water treatment methods and safe storage practices by 2020 (EMoH & WHO, 2015). However, the use of point-of-use water treatment methods is still low in Ethiopia.

Several clay pot-based, home treatment systems and devices are being used internationally by small, rural communities without bulk potable water services. Although some exploratory work relating to the use of these clay pots in other hygiene-based studies has been carried out in some African and Asian countries, little has been done to evaluate these units or to improve their functioning in order to empower rural households without water services to ensure that the water they bring into their homes from untreated sources is safe to consume. The Potters for Peace clay pot, for example, has been used in countries such as Nicaragua and Cambodia for some time, but the silver nanoparticles used as the anti-bacterial material is simply painted onto the surface that will lead easily leaching out of the silver colloid. Incorporating/impregnating silver nanoparticle throughout the pot may increase water treatment efficiency. This research therefore undertaken to optimize the bacterial removal efficacy and filtration rate of silver nanoparticle impregnated porous ceramic water filter pot made from local raw materials, which is ideally both more effective and less expensive than the ceramic units currently in use.

1.2. Statement of the problem

Waterborne disease are highly affecting the people living in developing countries including in Ethiopia. In response to the persistent problems associated with waterborne diseases worldwide, new strategies for safe water provision are gaining attention, including treating drinking water at the household level to reduce the ingestion of pathogenic microbes. Taken together, devices that can be used to treat water and/or prevent contamination of stored water in the home are referred to as household water treatment (HWT) or point-of-use (POU) technologies. These comprise a range of options that can enable individuals and communities to reduce microbial pathogens or chemical contaminants in collected water at the point of use, usually at the household level. POU technology

has the potential to fill the service gap where piped water systems are not possible, potentially resulting in substantial positive health impacts in developing countries (Brown & Sobsey, 2006).

Eminently, the efficiency and effectiveness of ceramic water filters are based on the type of clay material used, processing parameters and the type of filter design. One of the foremost vital parameters is porosity (Yakub et al., 2013). Whereas a lot of studies have focused on clay-based water filters, little or no work has been done in Ethiopia on local clay based material filters that provide safe water for human consumption hence reducing deaths and diseases caused by unsafe drinking water. This study investigates the incorporation of silver nanoparticles and also study the effects of processing parameters on locally available clay materials towards the design of porous ceramic pot water filter to enhance its disinfection efficacy. The porous ceramic water filter pots also used to filter large solid particulate materials beside killing microorganisms using silver nanoparticles. This will ensure that filtration and disinfection are achieved in one step instead of the usual two steps. It is envisioned that this system will be advantageous in terms of ease of operation, low cost (equipment, operation and maintenance), overall simplicity and excellent disinfection capability.

1.3. Objective of the study

1.3.1. General objective

The overall aim of this study was to investigate the incorporation of silver colloid/silver nanoparticle in the ceramic pot water filtration in order to enhance its disinfection efficacy as a point of use (POU) potable water filter and to optimize the filtration rate of the porous ceramic pot.

1.3.2. Specific objective

The specific objectives were:

- ✓ To investigate the incorporation of silver nanoparticle (AgNP) on the ceramic pot water filter and the characteristics of the silver nanoparticles impregnated ceramic water filter pot.
- ✓ To investigate and optimize the filtration and disinfection efficacy of the silver nanoparticle impregnated ceramic water filter pot using water with counted and prepared E. coli bacteria.
- ✓ To investigate and optimize the performance of the silver nanoparticle impregnated ceramic water filter pot in terms of disinfection efficacy and filtration rate.

1.4. Significance of the study

This research paper can be used for commercial stake holders and for health researchers including government companies who are working in water treatment and supply. Especially this research is very important for countries like Ethiopia which over the half number of the population did not get treated and safe water for drink. Therefore, this AgNP impregnated ceramic water filter pot can be used in the rural areas and some cities of the country to treat pathogens and some infectious disease.

By using this research, we can reach for peoples who did not get safe water drinking especially in rural areas with low cost of production to decrease due to water borne diseases. Researchers also use this paper for further researches in water treatment that can benefit the people in need.

1.5. Scope of the study

In this study, clay soil and sawdust will be pretreated (grinded and sieved), mixed and will be blended with water. The blended mixture then pressed and molded using materials like plastic water buckets on the way it can hold water to be filtered. The molded material/pot then dried by sun light for 14 days to remove moisture and fired in the kiln to burnout the burning materials (sawdust) so that the pore will be created which is permeable to water and to strengthen the pot. After firing, the filters are cooled and immersed in a solution containing silver nanoparticles (AgNP) and also demonstrated the disinfection property of the silver nanoparticle on the ceramic pot using prepared E. coli cell mass on water which is already had an estimated number or mass of E. coli.

CHAPTER TWO

2. LITERATURE REVIEW

2.1. The Need for Water Filtration

Access to a clean, safe and sustainable source of pure water is the crying need of the present century especially for developing countries like Ethiopia. Contamination of water with pathogenic microorganisms has detrimental effect on human health. The World Health Organization (WHO) estimates that over 4 million deaths per year, of which more than 1.5 million involve children under the age of 5, due to unsafe drinking water (Clasen, 2008). Several studies have shown that water is a source of various waterborne infectious diseases affecting many communities, mainly those in rural and undeveloped indigenous areas and consequently an estimated five million people lose their lives due to water-related disease each year (Fewtrell, 2014; Ds Lantagne et al., 2006; Murindababisha et al., 2018). Bacterial pathogens in water mainly causes gastrointestinal infections such as diarrhea, dysentery, typhoid shigellosis and human enteritis. The most common cause of illness and deaths in the developing countries is a watery diarrhea called cholera (Clasen, 2008) caused by a bacterial pathogen known as *Vibrio cholera*.

In Ethiopia 35% of the population didn't get improved or safe water for drinking according to the central statistics agency (CSA) and world bank report in 2017 (CSA, MOWIE, World Bank, UNICEF, 2017). From 60 to 80 percent of communicable diseases are attributed to limited access to safe water and inadequate sanitation and hygiene services. In addition, an estimated 50 percent of the consequences of undernutrition are caused by environmental factors that include poor hygiene and lack of access to water supply and sanitation. There are strong links between sanitation and under developing, and open defecation can lead to fecal-oral diseases such as diarrhea, which can cause and worsen malnutrition. Under five mortality death in Ethiopia is caused by Diarrhea which is the leading cause accounting for 23 per cent of all under-five deaths – more than 70,000 children a year (Wash, 2018).

Centralized water treatment facilities, like those found in cities and suburban areas in the developed world, are not feasible for many developing communities due to the large infrastructure investment. Alternatively, the WHO has suggested a decentralized approach of treatment in home immediately prior to consumption commonly referred to as point-of-use water treatment (Clasen,

2008; Ds Lantagne et al., 2006). Point-of-use water treatment technologies have the potential to significantly improve microbial quality of drinking water and reduce the risk of diarrheal disease and death, particularly in children (Clasen, 2008). A point-of-use water treatment technology must be effective with respect to removal and/or deactivation of waterborne pathogens under a wide range of water chemistries and must be simple to use to ensure long-term effectiveness and reduce risk of recontamination (Ds Lantagne et al., 2006).

Ceramic water filters, produced with local labor and materials, are an appealing point-of-use water treatment technology. Clay, sawdust and water are mixed and then molded into a pot shape. After that filter is then fired in a kiln, causing the clay to sinter into a ceramic and sawdust to combust that creates pore channels that allow water flow. Then quality testing is done and the filter is impregnated with a silver nanoparticle solution if we did not use silver colloid (silver nitrate solution) at the initial, where the silver acts as a well-studied antimicrobial agent without changing the taste, color, or odor of treated water. The ceramic filter is suspended inside a plastic bucket with a spigot on the bottom for personal use. Source water is poured into the ceramic filter, then the water percolates through to the lower reservoir, and clean water is dispensed through the spigot. The relatively small pore size (mean around 10 to 40 μm) of the ceramic filter helps remove turbidity and larger particles (D. S. Lantagne & Environmental, 2001).

2.2. Points-of-Use Water Treatment Systems/ Purification Methods

Many researchers(Clasen, 2008; Mattelet, 2006; Region et al., 2006) have proposed point-of-use water purification systems. These points-of-use technologies offer the advantages of being easily maintained and simple to use. The points-of-use technologies cover microbiological and/or chemical or physical water treatment including (Mattelet, 2006; Region et al., 2006):

- Disinfection (chlorination, solar disinfection (SODIS), solar pasteurization, UV irradiation with lamps, and boiling),
- Particle filtration (cloth fiber, ceramic filter, bio-sand and other slow sand filter technologies),
- Adsorption media (granular activated carbon, and activated alumina, clay)
- Combined system (combined flocculation/disinfection, filtration plus disinfection),
- Other approaches include plain sedimentation settling, safe storage, coagulation/flocculation with iron or alum salts and membrane processes.

2.2.1. Solar Water Disinfection (SODIS)

SODIS is one of household water treatment mechanism, which have been used for centuries in various parts of the world. It is a mechanism of exposing raw water to sun light for a minimum of 8 hours to kill bacteria's and other microorganisms using solar radiation. This can be achieved through synergistic effect of both UV-A light (320 - 400nm) and heat of solar radiation. It is reported that SODIS reduce faecal coliform and vibrio cholera to 99% in laboratory setting (Yilma et al., 2009). This method is suitable for treating small volumes of water (less than 5 liter), having relatively low turbidity of less than 30 NTU for the community lacking safe water supply (WHO, 2002).

2.2.2. Boiling

Boiling is the other oldest water treatment method of producing safe drinking water at household level. Boiling of untreated water for one to ten-minute can destroy disease-causing microorganisms found in water (WHO, 2002). According to (WHO, 2002), the method destroys all classes of waterborne pathogens such as viruses, bacteria, bacterial spores, fungi, protozoan and helminths ova. This method can be effectively applied to all types of water, including those with high turbidity and dissolved constituents (WHO, 2002) Boiling is commonly used in different part of the world especially during emergency such as outbreak of diarrhea disease. This method provides quick solution to purify drinking water at household level during the destruction of water supply systems caused by natural disasters such as hurricane, tsunami, earthquake and etc.

However, this technique is unsustainable both economically and environmentally in most developing countries. According to (Mintz, 1995) study to boil only 2 liters of drinking water would require 10 kilograms of firewood. Boiling on this magnitude would significantly affect environment by increasing deforestation pressures. On the other hand, alternative fuel sources available in these countries, such as kerosene and other fossil fuels, are often expensive.

2.2.3. Chlorination

Chemical disinfection is recognized as an effective and safe way to treat water at the point of use. There are a variety of different chemicals used but by far the most common in developing countries is chlorine and its compounds, usually bleach or sodium hypochlorite. Chlorine can be added either in liquid or in powdered form. Chlorine, if not handled properly or if the dose is too high can be

potentially harmful to health and so it is important that it is managed carefully and staff who are required to handle chlorine are well trained.

Chlorine can either be administered through a central supply system managed by trained government staff, the private sector or NGOs – examples would be either a piped water supply or tankered water, or it can be managed at the household level, preferably with fixed dose measures such as aqua tabs or sodium hypochlorite solution.

Given sufficient contact time, chlorine causes chemical reactions, which inactivate or kill microbiological contaminants in the water. It also oxidizes organic matter, manganese, and iron and hydrogen sulphide. Treatment with chlorine not only disinfects water at the point of use but the chlorine residual can protect water from recontamination for several hours afterwards.

Treatment with chlorine kills almost all bacteria and viruses but is not guaranteed to inactivate pathogenic parasites and chlorine works best in clear water. In turbid water it reacts with organic compounds, metals and other dissolved compounds, and the higher the turbidity the more quickly the chlorine is quickly used up, often before all pathogens have been killed. Turbidity makes it difficult to judge the concentration of chlorine that a quantity of water requires and so ideally turbidity of water should be tested and the concentration of chlorine calculated accordingly.

2.2.4. Bio-Sand Filter

Bio-Sand Water Filter (BSF) is another appropriate, effective and environmentally friendly water treatment technology to rural poor households (Mintz, 1995). BSF improves the biological and physico-chemical quality of drinking water through both mechanical and biological process. The filter was first used for water treatment in 1993 in Nicaragua (Donison, Kori S. (Kori Shay), 2004). The technology works on the basic principle of slow sand water treatment system. There are two types of BSF which is known in different parts of the developing countries. Those filter devices are Concrete BSF and Davnor plastic BSF model. Davnor Plastic Bio-Sand Filter is made of plastic material. This model is originally manufactured by water treatment technologies of Canada. It differs from concrete model mainly by its weight and materials cost. Particularly, light weight of the Source: DC, 2006 filter is the main distinguishing feature of the filter model compared to the concrete type. The concrete filter is heavy in weight since it is made of gravel and cement (Yilma et al., 2009).

2.2.5. Sedimentation

Sedimentation through storing water and allowing it to settle is an effective and low-cost natural sedimentation method to reduce water turbidity but it is not consistently effective in reducing microbial contamination; point of use treatment is also needed.

The effectiveness varies depending on the type of turbidity; most viruses, bacteria and fine clay particles are too small to settle out by simple gravity sedimentation. For this reason, it is usually recommended as a pre-treatment method, as treatments such as chlorination or UV radiation are known to work better on less turbid water.

Material and skills requirements are minimal. Two storage vessels are usually enough, one to store the sediment water and another in which to decant the clear supernatant into. Care should be taken not to disturb the sediment when decanting the water. It is essential that the solids are removed and the vessel cleaned on a regular basis.

2.2.6. Porous ceramic filters

Porous ceramic filters made of clay, carved porous stone and other materials have been used to purify water since ancient times (Brown & Sobsey, 2006). Modern ceramic filters are usually in the form of disks, pots or hollow cylindrical “candles” (Mattelet et al., 2006). A Ceramic candle water filter comprise of an upper and a lower bucket, with one or more clay cylindrical, hollow ‘candles’ screwed into the upper bucket which function as the filter. As the dirty water from the upper bucket passes through the candle, microbes and other suspended solids are removed, and clean water drips through into the lower bucket where it can be tapped off. The ceramic candle may be impregnated or coated with silver, which acts as a germicide and prevent microbial growth. According to Rivera-Sanchez evidence suggests that filters can be microbiologically very effective if they are correctly used and maintained (Rivera-sanchez et al., 2019).

Ceramic filters are fabricated from a variety of mineral media including different types of clay (kaolin, red terracotta, black clay), diatomaceous earth, or glass typically mixed with a fine combustible material such as flour, sawdust, rice husk (Mattelet, 2006; Nichols et al., 2007). The pore size and filtration properties of different filters depend on the materials and fabrication methods used.

Different types of ceramic filters are used to remove turbidity and microorganisms, chemical contaminants such as arsenic and iron, and even organic compounds which cause taste and odor (Lantagne, Quick, and Mintz, 2006; Rayner, 2009). Some ceramic filters are manufactured to contain activated carbon for organics removal. Carbonaceous material is incorporated into the filter element and converted to activated carbon by slow heating in the absence of air. This improves the filter performance especially in terms of taste and odor removal (Offringa, 2010; Wani & Kumar Sah, 2016). However, activated carbon can also provide a breeding ground for microorganisms which can negatively impact overall microbial removal.

The focus of this study is to assess and test filters which improve the microbial quality of the water. Removal of turbidity and microbes occur by straining or adsorption depending on the filter material, pore size and characteristics of the particles being removed. These filters are often also coated or impregnated with silver to provide them with antibacterial properties. This enhances bacterial removal/inactivation and also prevents biofilm growth on the element. Filters must be periodically cleaned to remove accumulated solids and restore flow rate. It is also imperative that filters are structurally sound as even small cracks will result in bypassing and contamination of the filtered water. Silver nanoparticle impregnated porous ceramic filters are therefore considered as an appropriate technology for developing countries. The basic raw materials are clay and a fine combustible material such as flour, sawdust and rice husk which is available almost anywhere in most of the parts of the world.

2.3. History of ceramic pot filter

Ceramic pot filters (clay pot filters) have been found to provide excellent clean water technology for household (or point-of-use) water purification in developing countries. They were originally developed in Guatemala in 1981 by Dr. Fernando Mazariegos. Potters for Peace and Resource Development International – Cambodia (RDIC) are the two main research institution which are American based institutions that promotes, develops clean water technology especially using ceramic pot filter. The two most common types of ceramic water filter are pot-type and candle-type filters. Ceramic filter systems consist of a porous ceramic filter that is attached to, or sits on top of a plastic or ceramic receptacle. Contaminated water is poured into a top container. It passes through the filter(s) into the receptacle below. The lower receptacle usually is fitted with a tap (Simonis & Basson, 2012).

2.4. How pot filters are made

Pottery is an ancient technology developed by humans during the period from 20,000 to 12,000 BC in Eastern Asia (Carli & Eagly, 2018). Little is known about how primitive humans developed and created ceramics, but the basic principle of wetting clay, then shaping and drying it into a shape to be heated to the point of vitrification is still present today (Gupta et al., 2018).

Pottery is the art of combining clay with water to create a material that is plastic enough to be shaped, but stiff enough to lose that shape when unsupported. As water evaporates from the clay, the plasticity drops and the material becomes stiffer. To further strengthen the material, it is slowly heated to over 900°C; each ceramic has its own required temperature (Halem, 2006). This heating step turns the ceramic into glass in a process known as vitrification, which strengthens the material and makes it non-porous.

Clay and combustible materials sourced locally could be used for the production of a ceramic pot filter (CPF). The process of production is the same with non-porous ceramic pots except using burnout materials to make porous after applying high temperature.

2.5. Global and national need for access to safe water

Safe and readily available water is important for public health, whether it is used for drinking, domestic use, food production or recreational purposes. Improved water supply and sanitation, and better management of water resources, can boost countries' economic growth and can contribute greatly to poverty reduction. According to world health organization and united nation international childrens fund report 2.2 billion people lack access to safely managed drinking water services and over half of the global population or 4.2 billion people lack safely managed sanitation services (UNICEF/WHO, 2019). From all of these 297,000 children under five die every year from diarrheal diseases due to poor sanitation, poor hygiene, or unsafe drinking water (WHO, 2019). Over 2 billion people live in countries experiencing high water stress and according to UNISDR 2019 report, 90 percent of natural disasters are weather-related, including floods and droughts.

Therefore, to decrease the death rate due to unsafe drinking water, the world needs to improve and increase the capacity of the availability of safe and clean water for drinking and other services especially in developing countries. When water comes from improved and more accessible sources, people spend less time and effort physically collecting it, meaning they can be productive

in other ways. This can also result in greater personal safety by reducing the need to make long or risky journeys to collect water. Better water sources also mean less expenditure on health, as people are less likely to fall ill and incur medical costs, and are better able to remain economically productive (WHO, 2019). With children particularly at risk from water-related diseases, access to improved sources of water can result in better health, and therefore better school attendance, with positive longer-term consequences for their lives.

2.6. Water quantity

Basic domestic water quantity needs can be divided into categories including water for drinking, cooking, hygiene, and other domestic purposes, including productive uses. Availability of sufficient water quantity close to the home is important for health, hygiene and quality of life; however, water quality is of primary importance when discussing drinking water. Drinking water needs can vary according to the water content of food consumed, manual labor performed and climatic conditions. In addition, men, children and women have varying needs. Even among women, needs vary when pregnant or lactating. In Table 2-1 suggested daily water requirements for hydration are presented. Since diets vary it is difficult to estimate the amount of fluid obtained from food. The following estimates are for hydration requirements, including fluid obtained from food (Lantagne et al., 2006; Rayner, 2009).

Table 2. 1: - Average Drinking Water Requirements

Category	Average conditions (liters)	Manual labor or in high temperatures (liters)	Pregnant or Lactating (liters)
Adult men	2.9	4.5	
Adult female	2.2	4.5	4.5 – 5.5
Children	1.0	4.5	

2.7. Water quality

Water can be contaminated both chemically and microbiologically. Of primary concern in efforts to reduce mortality and morbidity caused by infectious disease is the microbiological quality of water. Pathogens transmitted through contaminated drinking water include pathogenic bacteria (ranging about 0.5-3.0 microns in size), viruses (0.02-0.1 micron), protozoa (3.0-30 microns) and helminths (ova are about 45 microns). Although diarrheal disease can be transmitted through drinking water, it can also be transmitted via other faecal-oral routes. Transmission pathways

include consumption of contaminated food or drinks, poor sanitation and poor personal hygiene. Since water quality alone may not interrupt transmission of diarrhea, interventions often include increased water quantity, improved water quality, improved sanitation and the promotion of health and hygiene practices (Bank, 2017; Clasen et al., 2003).

2.8. Assessing water quality

Due to the difficulty of monitoring water for specific contaminants, water is examined for the presence of indicator organisms associated with faecal contamination. Commonly used indicator organisms (organisms used to measure treatment effectiveness) include total coliforms (TC), thermo-tolerant coliforms or faecal coliforms (TTC) and *E. coli*. Criteria for indicator bacteria as outlined by the World Health Organization are that they should be (WHO, 2005):

- ✓ Universally present in high numbers in human or other warm-blooded animal faeces
- ✓ Readily detectable by simple methods
- ✓ Should not grow in natural water

Some coliforms can grow and survive in water and are often present in the absence of faecal contamination. Therefore, the ‘total’ coliform count is not useful as an index of faecal contamination, however, it can be useful as an indicator of treatment effectiveness (Nichols et al., 2007; WHO, 2007). Thermo-tolerant coliforms are those of the total coliform group which are able to ferment lactose at 44 - 45°C. *E. coli* is often the predominant thermo-tolerant organism and is rarely found in the absence of faecal contamination. For this reason, *E. coli* is slightly more reliable as an indicator bacterium, however, other thermo-tolerant coliforms are also acceptable (WHO, 2006). Although *E. coli* has been established as the most suitable indicator by the WHO, it is also noted that the absence of *E. coli* does not ensure water safety since some pathogens are more resistant to some disinfectants. Therefore “verification may require analysis of a range of organisms, such as intestinal enterococci, (spores of) *Clostridium perfringens* and bacteriophages” (Lantagne et al., 2006).

In contrast to *E. coli* which are gram-negative bacteria, intestinal enterococci are gram-positive, which have a thicker cell wall. Intestinal enterococci are primarily of faecal origin and can be used as an index of faecal contamination. *Clostridium perfringens* are also gram-positive and resistant to UV irradiation, temperature, pH extremes and disinfection processes (WHO, 2007). Due to

their long survival times, they are not recommended for routine monitoring; however, they may be useful indicators for filtration effectiveness. *C. perfringens* should be removed by filtration processes designed to remove enteric viruses or protozoa (WHO, 2007). Bacteriophages (phages) are viruses which use bacteria as hosts and share many characteristics with enteric viruses. They are useful models to assess behavior, sensitivity to treatment and disinfection, however, they are not necessarily reliable as an index for faecal contamination since viruses have been found in water which tested negative for phages. There are two main types of phages: Somatic and F-RNA, the latter is better both as an index of faecal contamination and indicator for virus behavior (CSA, MOWIE, World Bank, UNICEF, 2017; WHO, 2007).

2.9. Drinking water quality guide lines in the world

Since different exposure levels might affect populations differently, the probability of infection is difficult to estimate based on water quality. The WHO guideline is that *E. coli* or thermo-tolerant coliform bacteria should not be detectable in any 100-ml sample of drinking water (WHO, 2006), however risk levels can be evaluated using a classification system for the microbiological quality of water presented in Table 2-2 based on WHO classification.

Table 2. 2: - Water Quality Risk Levels

Number of Thermal tolerant (faecal) coliforms or <i>E. coli</i> per 100 ml water sample	Risk level
0	Conforms to WHO guidelines
1 – 10	Low risk
10 – 100	Intermediate Risk
100 – 1000	High risk
1000+	Very high risk

2.10. Household water treatment and safe storage

A new set of technology known as household water treatment and safe storage (HWTS) or point-of-use (POU) water treatment has been developed and disseminated during the past two decades in an effect to improve the quality of drinking water in the developing world. It is commonly referred to as treating water and safely storing it at the household or community level. Household water treatment and safe storage (HWTS) often uses the same basic approaches of conventional

water treatment such as filtration, coagulation, and disinfection (boiling, chlorination, solar) on a smaller decentralized scale (Wong, 2014).

2.11. Silver impregnated Porous Ceramic pot filters and factors affecting the filtrations

2.11.1. Factors affecting filtration Efficiency

The factors that affect the efficiency of ceramic water filters include; porosity, turbidity of the water to be filtered, and the flow rate through the filter.

2.11.1.1. Effect of Porosity

Porosity is one of the main determining criteria that affect filter performance. High porosities in ceramic water filters is achieved by use of high percentages of burnout and also use of large particle sizes of burnout prior to firing. The most commonly used materials as burnout are carboneous materials. When the mass is fired, the carboneous matter burns out, leaving corresponding pore spaces so that the porosity of the fired mass is roughly proportional to the volume of carboneous matter added. The main combustible materials are hard wood sawdust, rice husks, naphthalene and occasionally fine ground coke, flour, corn husks (Vold, 1960) and others burn out materials (Nardo, 2005).

Petroleum waste products may also be used. However, they burn out at higher temperatures than wooden sawdust (Kaminskas and Ius, 2005). Wood consists of volatile oils and small quantities of mineral content. The mineral matter in wood consists mostly of salts of calcium, potassium and magnesium (Kaminskas and Ius, 2005). Ash is formed from mineral matter during combustion. The ash yield has a very small proportion of 0.1% to 1% by volume which has little effect on the porosity of the ceramic and during combustion the mineral ions oxidize and volatilize or form particulates (Ragland, Aerts, and Baker, 1991).

The size of the pores is also important in controlling the flow rate and the level of contaminant removal of the filter. Filters showing large pore sizes will not be efficient at removing turbidity and /or bacteria from water as compared filters having small pore sizes. However, as filters flow rate increases with filter pore size, these filters will allow the collection of more water per hour (Mattelet, 2006). For high filtration rate, the porosity should be high but the pores should be very small (Nano-scale) in diameter for effective filtration. The underlying principle behind the water

filter is the amount and the size of combustible (sawdust) which have been milled and screened, combust in the firing furnace/kiln leaving a network of fine pores.

The volume porosity of the filters can be determined by a number of different methods including:

(a) Ultrasonic pulse velocity (UPV) measurements

This is a nondestructive method which is simple, accurate and can detect size and defects (Hall and Hoff, 2010). The relation relating ultrasonic pulse velocity to porosity is

$$VL = VLO (1 - MP) \dots\dots\dots 2.1$$

Where, VL is the ultrasonic pulse velocity in the porous material, VLO is the ultrasonic pulse velocity of the filter at zero porosity, M is a constant which is a function of Poisson's ratio

Rearranging the above equation gives

$$P = \frac{\Delta V}{M} VLO \dots\dots\dots 2.2$$

Where, ΔV is the change in ultrasonic pulse velocity of the specimen

(b) Density method

Porosity is defined in terms of bulk density according to the relation (Volfkovich, Sakars, and Volinsky, 2005).

$$\rho_B = P\rho_a + (1 - P)\rho_s \dots\dots\dots 2.3$$

Where, ρ_B is bulk density of sample, ρ_s is density of the pure solid, ρ_a is the density of air which is much smaller than ρ_B .

Other methods for determining porosity include gas expansion method, optical method, mercury standard porosity and the most recent is the automated standard porosimeter (ASP) which is based on the laws of capillary equilibrium (Volfkovich, Sakars, and Volinsky, 2005).

2.11.1.2. Water Turbidity

Turbidity is an indicator in assessing the suitability of water for human consumption (UNICEF and WHO, 2004). It stimulates the growth of bacteria and other water pathogens (UNICEF and

WHO, 2004). Along with E. coli, pH and chlorine residual, turbidity is one of the key parameters of microbial water quality. It is also used to measure the effectiveness of water filtration.

High organic concentration and/or many suspended particles such as clay, silt, planktons, and other microscopic organisms and chemical precipitates will slow the flow rate of water by progressively clogging the ceramic pores. This will affect the rate of filtration (Mattelet, 2006). Turbidity is measured by determining the degree of light scattering by particulates present in the sample (Miller, 1997). Water to be treated by use of ceramic filters should have a maximum pre-treatment turbidity of 15-20 NTU (van et al., 2006) to avoid the clogging of pores. (UNICEF and WHO, 2004), recommends turbidity of one Nephelometric Turbidity Unit (NTU) and up to 5 NTU.

Light scattering by transparent isotropic media is accurately described by a macroscopic fluctuation theory first formulated by Einstein (Miller, 1978). Turbidity Γ , is dependent on fluctuations in the refractive index (or the di-electric constant) in volume, v , according to the equation;

$$\Gamma = \frac{[32 \cdot \pi^3 \cdot v \cdot n^2 (\delta n)^2]}{3 \cdot \lambda_0^4} \dots\dots\dots 2.4$$

Where; λ is the wavelength of the incident radiation in vacuum and $(\delta n)^2$ is the fluctuation average in the reflective index n

In an absorbing media, the refractive index is complex and therefore $(\delta n)^2$ can be replaced by, $(\delta n)^2 + (ac)$. Where, ac is the imaginary part of the complex refractive index?

The absorption coefficient ac must be much smaller than the real part if one is to be able to detect any scattered light (Miller, 1978).

For effective filtration, the filter membrane needs regular cleaning with a specified scrubbing brush to remove deposits. Pre-treatment of water is also an important method of controlling fouling of the filter membrane (Contreras et al, 2009).

2.11.1.3. E. coli removal

Within the filter, multiple pores throughout the membrane are interconnected, forming channels that allow for the passage of water. One method of pathogen removal is to block particles and organisms that are larger in size than the pores from flowing through the outermost membrane

layer (Doulton, 2009). It is possible that they will adsorb on to the ceramic or become blocked when larger particles plug up the pores.

2.11.1.4. Flow rate

Water flow rate is given by equation (2.5) and it is also dependent on pore characteristics (Hagan et al., 2008).

$$Q = \frac{r^3 \Delta P}{3 \mu} \left[1 + \frac{8L}{3\pi r} \right]^{-1} \dots\dots\dots 2.5$$

Where r the radius of the pore, μ it is the coefficient of dynamic viscosity, L is the thickness of the filter, Q is the flow rate, and Δp is the pressure drop

The larger and more connected the pores, the easier it is for water to flow through the membrane. Adjusting pore size, will affect both flow rate and microorganism removal. Filter design variables, that are pore related and could potentially be manipulated to affect flow rate include the type of combustible material, the amount of time the clay/combustible material is mixed, the thickness of the ceramic membrane, the size of combustible material particles, and the proportion of combustible material to clay.

2.11.2. Factors affecting flow rate

Porosity and permeability are important in determining the rate of fluid flow through the filter. The filtration rate through the porous filter element (Figure 2.1) was assumed to follow Darcy’s law,

$$v = \frac{q}{A} = -\frac{k}{\mu} * \frac{dP}{dL} \dots\dots\dots 2.6$$

Where; v : apparent flow velocity, cm/s

q : Flow rate, cm³/s

A : cross sectional area, cm²

L : System length, cm

dP/dL : Differential pressure in L-direction, atm

k : Permeability, Darcy and μ : Fluid viscosity at system T, cp

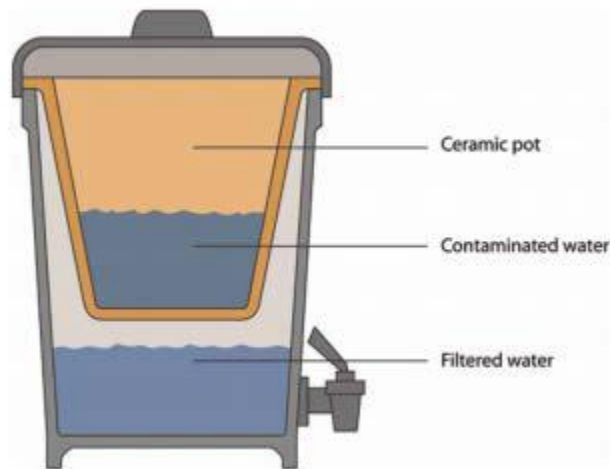


Figure 2. 1: - Typical setup of Silver nanoparticle impregnated porous ceramic filter (Gardner, 2017)

The pressure difference between the surface of water in the top bucket and the inner surface of the disc ceramic filter is equal to the hydrostatic pressure of the water. For the flow through the bottom, the difference in pressure from the inside bottom surface to the outside bottom surface is equivalent to the hydrostatic pressure of the fluid. The volume V , of the water at any given time was measured using a measuring cylinder. This volume together with the area, A , of the inside bottom of the top receptacle were used to determine the height, $h(t)$ of the water above the filter at any given time using Equation (2.7).

$$V = \frac{1}{3} * A * h(t) \dots \dots \dots 2.7$$

2.11.2.1. Thickness (tb) and Surface Area of the pot

The thinner the thickness of the porous ceramic pot, the higher the flow rate will be for a given water head (H) as can be seen in equation (2.7). With a thin ceramic device, flow rate increases but the level of water microbiological and turbidity removal will decrease. By increasing the thickness, the tortuosity of the ceramic element increases, therefore it will retain more particles (Region, Mattelet, and Whittle, 2006). The flow rate also increases faster for a given increase in surface area since Q is proportional to the radius squared.

2.11.2.2. Differential pressure (ΔP)

Differential pressure (ΔP) is the difference between the pressure in the system before the fluid reaches the filter (upstream) and the pressure after the fluid flows through the filter (downstream pressure) (Mattelet, 2006). In a constant flow application, the differential pressure increases as the filter begins to clog. While the water height decreases with time above the filter, the water pressure becomes smaller and therefore less water will flow through the filter. It is therefore important to maintain a certain level of water head above the filter by filling the upper vessel of the filter with water.

$$\Delta p = \rho * g * h(t) \dots\dots\dots 2.8$$

Volumetric flow rate is dependent on the pore size of the filter, membrane surface area, and the height of the water in the filter. By use of the law of conservation of mass which states that the mass of an isolated system remains constant over time (Wald, 2012).

$$Ra = Ri - Ro \dots\dots\dots 2.9$$

Where, R_i is rate of mass in, R_o is the rate of mass out and R_a is the rate of accumulation of mass.

If the filter is filled up with water and begins to filter the water without adding any more water to the top container, then we can say that the rate of mass in is equal to zero in the above relationship.

$$So - Ri = Ra \dots\dots\dots 2.10$$

This will give us the equation that states that the density of the fluid ρ multiplied by the volumetric flow rate of the fluid out of the vessel Q equals the rate of accumulation of the liquid outside the filter with respect to time (Wald, 2012),

$$-\rho Q = dm/dt \dots\dots\dots 2.11$$

Where m is the mass of the liquid and $\rho = 1000 \text{kg/m}^3$

Taking the mass of the liquid accumulation outside the filter equal to density ρ times the volume V in milliliters of the liquid;

$$m = \rho * V \dots\dots\dots 2.12$$

By integrating the circular cross-sectional areas with respect to the height (z) of the water above the filter we can find the volume at any height:

$$V(z) = \int_0^h A dz \dots\dots\dots 2.13$$

Where $A = \pi r^2$.

2.11.2.3. Velocity of Water flow

Darcy’s law states that the flow speed through a porous medium is directly proportional to the pressure gradient through that medium and to the square of the characteristic size of the pore spaces of the medium, and inversely proportional to the viscosity of the medium (Wald, 2012).

$$v = C * \Delta P \dots\dots\dots 2.14$$

Where, v is the velocity of water flow, C is the proportionality constant which depends on the size proportion of the porous medium as well as on the particle shape and packing, Δp is the change in pressure

$$\Delta P = P_t - P_{atm} \dots\dots\dots 2.15$$

Where, P_t is the total fluid pressure and P_{atm} is the atmospheric pressure

The total fluid pressure at any point of the water level in the filtration container is equal to the atmospheric pressure pushing down on the fluid above the surface of water in the container plus the pressure of water above the filter membrane (Wald, 2012). Therefore,

$$P_t = P_{atm} + \rho g * (h - z) \dots\dots\dots 2.16$$

Where, g is the acceleration due to gravity, h is the height of water in the container and z is the change in height while the water is flowing out of the filter

Subtracting atmospheric pressure from total pressure yields

$$P_t - P_{atm} = \rho g * (h - z) \dots\dots\dots 2.17$$

Using equation (2.15) with (2.17) we see that

$$V = C \rho g * (h - z) \dots\dots\dots 2.18$$

If we combine density, acceleration due to gravity and the proportionality constant into the coefficient of volumetric flow rate $a = C \rho g$ we obtain

$$V = a * (h - z) \dots\dots\dots 2.19$$

Equation (2.19) is clear evidence that velocity is proportional to the difference in pressure of water above the disk filter. If the velocity increases the flow rate will also increase.

2.11.2.4. Strength of the membrane

The strength of a ceramic material is inversely proportional to the porosity of the filter (Nyongesa and Aduda, 2004) given by the relation.

$$\delta = \delta_0 * e^{-bp} \dots\dots\dots 2.20$$

Where δ is the flexural strength, δ_0 is the flexural strength at zero porosity and b is a constant. The porosity of the filter is affected by amount of sawdust in the mixture. At very high percentages of sawdust there will be more voids created in the filter membrane and the filter might become very weak. On the other hand, if the amount of sawdust is very low, then the filter will be considered very strong but at the same time the filtration rate will be low and hence the efficiency will reduce. An optimal clay to sawdust ratio needs to be achieved in order to maintain an optimal filtration rate that will give a strong filter to support the hydraulic pressure above the filter and again an optimal ratio that will give an effective filter in terms of *E. coli* removal and also filtration rate. Optimal strength will make the filter to last for a long time without breaking.

2.12. Antibacterial mechanism of silver Nano particle

Silver ions and nanoparticles show a wide spectrum biocidal activity. Ceramic filters are often coated with silver nanoparticles to improve microbial removal and prevent biofilm development on the filters. Silver has a long history of use as an antimicrobial agent in water treatment and in other application such as an environmental biocide and ingredient in medications (Lantagne et al., 2006) however, the mechanisms involved are still not well understood. Feng et al., 2000 and Lantagne et al., (2001a) identifies four groups of mechanisms which have been proposed in the literature:

- ✓ Silver reacts with thiol (-SH) groups in structural groups and inactivates vital functional enzymes found that electrically generated colloidal silver inhibited 16 bacterial species including *E. coli* at 1.25 $\mu\text{g}/\text{m}\ell$ colloidal silver and killed them at 10.5 $\mu\text{g}/\text{m}\ell$. Mammalian cells were not affected.

- ✓ Silver produces structural changes in bacterial membranes. Feng et al. (2000) observed structural changes in cell membrane involving silver reacting with Sulphur containing groups. This was thought to interfere with cell respiration and permeability.
- ✓ Silver interacts with nucleic acids to disrupt cell function. (Feng et al., 2000) found that DNA loses replication ability after microbes have been treated with silver and suggested that this may be due to the tendency of silver to react with phosphorous groups.
- ✓ Silver deposited on inert surfaces exhibits a strong catalytic reaction with oxygen which results in bacteria and viruses being killed on contact (Charles, 1993). This mechanism is thought to be of particular relevance to silver impregnated filter. (Uri and Zhang, 2013), found that the rate of the catalytic reaction depended on the size and dispersion of the silver on the surface and the oxygen concentration.

2.12.1. Syntheses methods of Silver Nanoparticles

2.12.1.1. Physical Approach

In physical processes, silver nanoparticles are generally synthesized by evaporation condensation, which could be carried out using a tube furnace at atmospheric pressure. The source material within a vessel centered at the furnace is vaporized into a carrier gas. However, the synthesis of AgNP's using a tube furnace has several limitations, because a tube furnace needs a large space, consumes a large amount of energy while increasing the environmental temperature around the source material, and needs a lot of time to achieve thermal stability. A typical tube furnace needs power consumption of large amount of kilowatts and a preheating time of several hours to attain a stable condition of operating temperature (Qin et al., 2010; Thi et al., 2019). The physical synthesis of AgNP's usually utilizes the physical energies to produce AgNP's with nearly narrow size distribution. The physical approach can enable producing large quantities of AgNP's samples in a single process. This is also the most useful method to produce AgNP's powder. However, primary costs for investment of equipment is large and should be considered (Natsuki, 2015).

2.12.1.2. Photochemical Approach

The photo-induced synthetic methods have also been developed. For example, Huang and Yang synthesized AgNP's via photoreduction of AgNO₃ in layered inorganic clay suspensions, which used as stabilizing agent that prevent nanoparticles from aggregation (Huang et al., 2016).

Irradiation disintegrated the AgNP's into smaller size with a single mode distribution until a relatively stable size and diameter distribution were achieved. However, in this method, the equipment's with high cost and experimental environment are required (Haider & Kang, 2015).

2.12.1.3. Biological Approach

In recent years, biosynthetic methods using natural reducing agents such as polysaccharides, biological microorganism such as bacteria and fungus or plants extract, i.e., green chemistry, have emerged as a simple and viable alternative to more complex chemical synthetic procedures to obtain AgNP's (Desai et al., 2012). Bacteria are known to produce inorganic materials either intra- or extracellularly. This makes them enable bio factories for the generation of nanoparticles like gold and silver. Mainly, silver is well known for its anti-bacterial properties (Lagashetty et al., 2019). The biological silver nanoparticle synthesis method gives a wide range of resources for the synthesis of AgNP's, and this method can be considered as a method of nanoparticles synthesis with advantages over conventional chemical routes of synthesis and as an environmentally friendly approach as well as a low-cost technique. However, it is difficult to obtain a large quantity of AgNP's by using biological synthesis method (Haider & Kang, 2015).

2.12.1.4. Chemical Approach

Besides those methods described above, chemical synthesis is the most common method because of its practicality and the need for simple equipments for operation. Control over the growth of silver nanoparticles is required to obtain nanoparticles of small size with a spherical shape and uniform distribution in diameter. It is well known that silver nanoparticles can be produced by chemical reaction at low cost and in maximum yield (Vega-Baudrit et al., 2019). Generally, the chemical generation process of AgNP's in solution usually needs to undertake the following three main components: (1) metal precursors, (2) reducing agents and (3) stabilizing /capping agents. The formation of colloidal solutions from the reduction of silver salts involves two stages of nucleation and subsequent growth. It is also revealed that the size and the shape of synthesized AgNP's are strongly dependent on these stages (Jawaad et al., 2014).

Generally chemical methods of synthesising silver nanoparticles are more advantages than the other methods due its capability giving higher yild and ease of operation.

CHAPTER THREE

3. MATERIALS AND METHODS

3.1. Materials Used on this Experiment

3.1.1. Raw materials

The major raw materials used in this study were clay soil, saw dust, E. coli and silver nitrate. The clay soil was collected from North Gondar around small city called Enfranz where there is a high clay soil used for production of clay pots, charcoal cooking stoves which is locally called ‘fermelo’ and injera baking stoves called ‘mittad’. There are peoples who are living there by working as a craftsmen and craftswomen who are coming from Jew family. The clay soil collected from there were grinded using a Jaw crusher and sieved using 1000 μ m size mesh to make smaller particle which is less than 1000 μ m for investigation.

The burnout material used in this study was sawdust which is a mixture of different hard wood trees used to make furniture production that was collected from local furniture manufacturers in Addis Ababa. Sawdust was selected as a burnout in this study because it gives a homogenous mixture with clay, unlike with any other burnout material like coffee husks and rice husks and also because of sawdust has a low ash content than coffee husks and rice husks (Klarman, 2009). The sawdust found from wood materials also consists of volatile oils and small quantities of mineral content. The mineral matter in wood sawdust consists mostly of salts of calcium, potassium and magnesium which are volatiles and decreases the amount of ash content and increases the porosity of the ceramic pot filter. In addition to this, hard wood sawdust is preferred to soft wood sawdust because it does not bloat as much as sawdust from other woods, thus leading to filters having more uniform pores and fewer defects (Klarman, 2009).

Sawdust collected from local furniture manufacturers then sieved using mesh wires from 200 μ m to 800 μ m. Mesh size from 200 μ m to 400 μ m, from 400 μ m to 600 μ m and from 600 μ m to 800 μ m are used for the fabrication of different silver nanoparticle impregnated porous water filter ceramic pots. The other chemical and bacteria used as raw material are silver nitrate and E. coli. Silver nitrate was used as anti-microorganism agent by impregnating in to the porous ceramic pot whereas E. coli was used to test the efficiency of fabricated silver nitrate impregnated porous ceramic pot.

3.2.1.3. Particle size analysis

Sawdust particle size is the major factor to determine the porosity of ceramic water filters. Therefore, to determine the particle size of sawdust, sieve analysis method was employed.

Sieve analysis

Sieve Analysis involved the use of four sieves to distribute different particle sizes in the sawdust used in this study. The sieves were thoroughly washed in clean water and dried in the oven. The mass of each sieve as well as the bottom pan to be used in this analysis were measured. The mass of the sawdust was also measured. The sieves were then assembled in descending order of sieve sizes (size 800 μ m sieve at top) and (size 200 μ m sieve at bottom). The pan was placed below sieve size 200 μ m. Five hundred grams of the sawdust sample was then poured into the top sieve. The sieve stack was placed in mechanical shaker and shaken for 10 - 15 minutes.

The stack was removed from the shaker and the mass of each sieve with its retained sawdust recorded and also sieved all the samples using the same procedures. The mass of the bottom pan with its retained fine sawdust was recorded. The mass of sawdust retained on each sieve was obtained by subtracting the weight of the empty sieve from the total mass of the sieve and the retained sawdust's. The percent retained on each sieve was obtained by dividing the mass retained on each sieve by the original sample mass. The percentage passing also known as percent finer was obtained by subtracting the percent retained on each sieve as a cumulative procedure starting with 100%. The mass of sawdust retained at the top of 800 μ m sieve and the mass of sawdust passed through 200 μ m sieve are discarded since not using these samples for the present experiment. The screen/particles sizes that passed through 800 μ m, 600 μ m, and 400 μ sieve sizes are used for the fabrication of ceramic pot water filters.



Figure 3. 1: - Red clay soil and sawdust materials used for making CWFP

3.2.2. Preparation of silver nanoparticle (AgNP) using Sodium borohydride as reducing agent

3.2.2.1. Procedure for the synthesis of silver colloid (silver nanoparticles)

The synthesis of silver nanoparticles was prepared by chemical synthesis method using cold process. 5gm of Silver nitrate (AgNO_3) of 0.01 M and sodium borohydride (NaBH_4) of 0.02 M each containing 0.01 gm of sodium dodecyl sulphate (SDS) was prepared in conical flask with deionized water. Sodium borohydride was used, due to its strong reducing agent and also to increase the speed of the reaction. The prepared NaBH_4 solution was cooled in an ice bath for 20 minutes. The synthesis process is shown in process flow sheet on figure 3.1. Two solutions were prepared. Solution A of 250 mL was prepared by adding silver nitrate and SDS and solution B of 750 mL was prepared by adding sodium borohydride and SDS. A total concentration of 211 mg/L of silver nanoparticle was synthesized. The change of the colorless AgNO_3 solution to pale yellow was the indication for the formation of colloidal Ag nanoparticles. To confirm the formation of silver nanoparticles and the progress of reaction, the UV-Vis spectra analysis was done (Leersnyder et al., 2019).

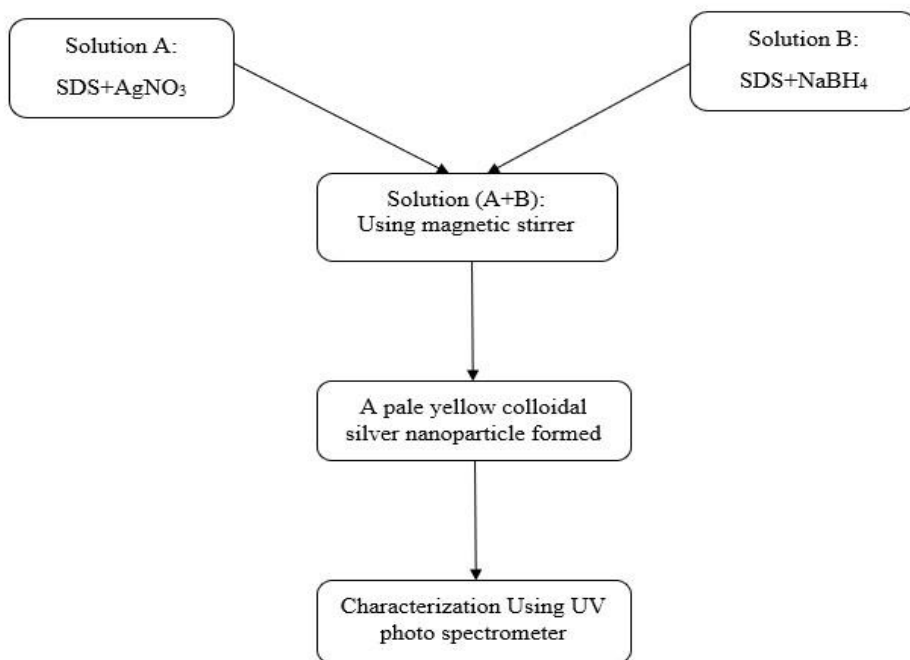


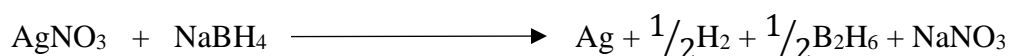
Figure 3. 2: - Synthesis of colloidal silver nanoparticles from silver nitrate using chemical reduction

Silver nanoparticles can be synthesized by various chemical methods: Reduction either by using Citrate, Sodium Borohydride or polyvinylepyrrolidone (PVP) and ion implantation synthesis. Amongst these, Sodium Borohydride was used for reduction of silver nitrate and stabilize the silver nano particles. Sodium Borohydride being a strong reducing agent used to maximize yield and to save time since the reaction is fast. An ice bath facilitates to slow down the reaction rate and eliminates secondary reactions (Banne et al., 2017).

3.2.2.2. UV-Vis Spectro photometer test

UV-visible spectroscopy is one of the popular characterization techniques to determine silver nanoparticle formation and its properties. And also, it is known that the spectrum surface plasmon resonance of nanoparticles is influenced by the size, shape, interparticle interactions, free electron density and surrounding medium, which indicates that it is an efficient tool for monitoring the electron injection and aggregation of NPs. UV-VIS spectroscopy was performed by SPECTRO UV-VIS double beam PC 8 Scanning Auto cell UVD-300 spectrophotometer at AAiT biochemical laboratory. The absorption spectrum was recorded from 200 nm to 800 nm.

The present of lone pair electrons in sodium borohydride (NaBH₄) are responsible for complexation as well as reduction of silver ions to silver nanoparticles. The reaction process that shows the synthesis of silver nanoparticle is shown below.



Even though there are lots of mechanisms of synthesizing silver nano particles, using sodium borohydride as a reducing agent and SDS as stabilizing agent was the easiest and the more effective methodology.

3.2.3. Fabrication of silver colloid impregnated ceramic pot

Silver nanoparticle/colloid impregnated porous clay ceramic water filter pots were fabricated using clay plus by varying mass composition of sawdust in the ratios of 85:15, 90:10, 95:5 which is used to know the impact of mass percentage ratio of sawdust on filtration and bacterial removal efficiency of the porous ceramic filter. Clay soil and saw dust were dry mixed well to keep uniformity and to distribute sawdust evenly with the clay soil. Then 0.5 liters of water was added per 1Kg of mixed clay and sawdust to make the dough. When the amount of water is less than 0.5 liter, the dough was too dry to mold the pot whereas when the amount of water added is higher

than 0.5 liter, the pot molded did not keep its shape (the mixture is too wet to make the pot filter). The mixing of these components was done manually by kneading and squeezing method as commonly used by potters. Wet mixing took 10 more minutes per addition of a drop of water before the dough was ready for pressing to ensure uniformity of the dough (Daniele Lantagne et al., 2017).

The mixture was then formed into balls of mass of 410.0 ± 0.5 g each from the bulk. This mass of the dough was enough to produce a ceramic pot filter of 1.0 ± 0.2 cm in thickness after pressing and about 0.6 ± 0.20 cm after drying and firing. The balls were in excess of 20 grams to ensure that air bubbles were pressed out of the walls of the filter in the molding press. The weighed portions were then formed into cylindrical shape at a loading pressure of 140 ± 5 kPa using a hydraulic press found at Mechanical lab of Addis Ababa University institute of Technology (AAiT).



Figure 3. 3: - Images of porous ceramic pots

The filters were left to dry in the laboratory for a maximum of 14 days at a room temperature of 20 °C before the firing process began. After drying, the molded filters were fired in an electric furnace at a temperature of 900 , 1000 and 1100 °C. This involved the preheating of the filter pots to 100 °C, and then held at that temperature for about 1 hour in order to allow ordinary water to escape and preventing cracking due to unbalanced energy before heating to 550 °C for 3 hours (to burn off the sawdust) followed by heating selected filters to the firing temperature of 900 , 1000 and 1100 °C and exposed at that temperature for five hours. Thereafter the furnace was switched off and left to cool to room temperature. Four filter pots were vertically stacked in the furnace with

a space of about 2 cm between one filter and the other. This was to ensure uniform distribution of the heat and also to ensure that the carbons inside the filters burn off completely to prevent cracking of the filters.

After firing and cooling the porous cylindrical ceramic water filters were submerged for 60min (to ensure time for AgNP's thoroughly impregnated) in a 211 mg/L colloidal silver (AgNP) solution which is already made and left to dry on an open surface for 2 day at room temperature before starting bacterial removal tests.

3.2.3.1. Scanning Electron Microscope (SEM) Analysis

Field emission Scanning Electron Microscope (SEM) was conducted at Addis Ababa Science and Technology University to check the impregnation of silver nanoparticles (AgNP) on the surfaces and inside the pores of the porous ceramic water filter pots. The sample was taken by abrading the surface of porous ceramic pot using glass paper, after immersing the porous ceramic water filter pots for 60min into the solution of AgNP with a concentration of 211mg/L and left to dry at room temperature for 2 days so that the AgNP settled and stick on to the porous ceramic water filter pots.

3.2.4. Measurement of physico-mechanical properties

During the study of silver nanoparticles impregnated ceramic water filter pot, different physico-mechanical properties of the porous ceramic pot and filtrate were determined.

3.2.4.1. Volume porosity

The apparent porosity of the AgNP impregnated ceramic water filter pot (CWFPs) was determined as the amount of water absorbed by the sample using the Archimedes Immersion Technique (Christopher et al., 1992) that involved boiling the samples in water for three hours.

The amount of water absorbed (percentage water absorption, %WA) was determined using the equation 3.2 where W_s and W_d are the saturated and dry sample masses, respectively.

$$\%W = \frac{W_s - W_d}{W} * 100\% \dots \dots \dots 3.2$$

The apparent porosity was calculated using equation (3.3)

$$P = \frac{\text{amount of water absorbed}}{\text{density of water}} = \frac{W_s - W_d}{W_d * \rho_w} \dots \dots \dots 3.3$$

3.2.4.2. Determination of flow rate

Three flow rate tests were performed on each of the fired ceramic pot water filters. The filter elements were first immersed in water and soaked for about 12 hours to ensure enough saturation of filters at the beginning of the test and also to remove internal air bubbles. Enough saturation of water enables continuous flow of water through the pores of the porous ceramic water filter pots.

The ceramic pot filter was assembled with receptacle together to collect the filtered water. The bucket containing the filter was then filled with the E. coli contaminated water to the brim, and the timer was started for one hour. The flow rates were then measured through all the ceramic water filters one at a time.

Flow rate was calculated using the relation (3.4).

$$flowrate = \frac{volume\ collected}{time\ elapsed} \dots\dots\dots 3.4$$

At the start of each experiment, the bucket with water saturated silver colloid impregnated CWFPP was filled completely with approximately 500 ± 0.5 ml of water and covered with lid to prevent evaporation which may lead to reduction of mass. As the water went through the porous filters for one hour, the filtered water was transferred from the receiver bucket to a measuring cylinder where its volume was measured.

3.2.5. Media Preparation and E. coli Removal Experiment

3.2.5.1. Media Preparation

Microorganisms depend on a number of factors such as nutrients, oxygen, moisture and temperature to grow and divide. In the laboratory, except for the above factors, the culture medium should be sterile and contamination of a culture with other organisms should be prevented. A microbiological culture medium must contain available sources of hydrogen donors and acceptors, carbon, nitrogen, sulfur, phosphorus, inorganic salts and, in certain cases, vitamins or other growth-promoting substances (Lessard, 2013).

To inoculate E. coli strain different media's can be used. For this research 5gm yeast extract, 5gm salt, 12gm agar and 10gm of peptone 140 was used per one liters of distilled water which then sterilized for 15min at 121 °C and allowed to cool at room temperature. Then E. coli K12 strain stroked on solidified nutrients on the petri dishes and incubated overnight for 24 hours at 37 °C.

Then the inoculated *E. coli* on the petri dishes transferred to a broth media which was prepared using 13gm of nutrient broth with one liters of distilled water and obviously sterilized for 15 minutes at 121 °C. The broth then incubated for another 24 hrs. and then prepared for serial dilution and another incubation on the solid media to determine the amount of *E. coli* in CFU/100ml for *E. coli* removal experimental test.



Figure 3. 4: - Images of inoculated *E. coli* bacteria on Petri dishes

3.2.5.2. *E-coli* removal experiment

The silver colloid impregnated ceramic pot filter were firstly tested using counted and prepared *E. coli*. 3.82×10^8 amount of prepared and counted *E. coli* was mixed with peptone buffered distilled water and allowed to pass through AgNP impregnated porous ceramic water filter pots. The amount of *E. coli* passed through CWFP were counted by taking 100ml of sample (the number of colonies forming units per 100ml) and compared with the WHO standard. All the tests were done using antiseptically sterilized materials.

The second test were done using tap or surface water collected from swamp surface at Kebena River. Water was collected from stagnant surface which contaminated with different coliform were filtered using the CWFP membrane and tested for indicator organism *E-coli*. All the equipment's used in this technique were put in boiling water to sterilize them each and every time in use. 100 ml sample of the effluents from all the filters were put in various petri dishes and placed in the incubator at 37 °C for 24 hours. The number of colonies forming unit (CFU) was counted under magnifying glass and expressed as CFU/100 ml. The indicator organism's level in water sample was expressed as the number per 100 ml and the number of the indicator organism's in water tested was determined using the relation (3.5).

$$Io = \frac{y}{mls} * 100 \dots\dots\dots 3.5$$

Where; *Io* is indicator organism found per 100ml of sample, *y* is indicator organism counted and *mls* is millimeters of sample per 100 ml. The data presented in log reduction calculation based on the formula below

$$\text{Log reduction} = \log_{10}\left(\frac{A}{B}\right) \dots\dots\dots 3.6$$

Where A = number of colonies before treatment and B = number of colonies after treatments

3.3. Experimental design for AgNP impregnated Porous ceramic pot used to treat bacteria on tap water

In this work AgNP impregnated porous ceramic pot was produced to treat bacteria and other microorganisms on tap water. Experimental data analysis was done using Design- Expert 12.0.3.0 software. The experimental design selected for this study was response surface methodology, three-level-three-factor Box-Behnken Design (BHD) and the response variable measured were the percentage of microorganisms removed and rate of filtration.

Three independent variables were studied including particle size of sawdust, ratio of sawdust to clay and the burning temperature of porous ceramic pot. The independent variables interaction effect was analyzed to obtain the optimum point of bacterial removal efficiency and filtration. Seventeen experimental runs were conducted to find the optimum point and triplicate experiments are done at the optimum points. After seventeen experiments were done, the data was statistically analyzed using Design-Expert Software 12.0.3.0 to obtain a suitable model equation for the percentage removal efficacy of bacteria and the optimum filtration rate of AgNP impregnated porous ceramic pot as a function of the independent variables.

Table 3-1 lists the range and levels of the three independent variables studied. The lower and higher levels are chosen by considering the maximum and minimum points of ceramic pot productions systems and based on some literatures.

Table 3. 1: - Independent variables and levels used in the BHD

Variables	Units	Levels		
		-1	0	1
sawdust amount used	%	5	10	15

Particle size of sawdust	μm	(200 – 400) = 1	(300- 400) = 2	(400 – 500) = 3
Furnace temperature	°C	900	1000	1100

Below in Table 3-2 the complete experimental design matrix of BHD for the factorial design was shown. The order in which the runs were made was randomized to minimize systematic errors. Sawdust particle size that are used in certain amount of ranges represented by simple numbers (1, 2 and 3) as described on the above table.

Table 3. 2: - The complete experimental design matrix

Runs	Actual factors		
	% sawdust amount used	Particle size of sawdust (μm)	Temperature (°C)
2	5	200 – 400	1000
5	5	400 – 600	900
10	5	400 – 600	1100
17	5	600 – 800	1000
1	10	200 – 400	1100
3	10	400 – 600	1000
6	10	600 – 800	1100
7	10	400 – 600	1000
8	10	400 – 600	1000
12	10	400 – 600	1000
13	10	600 – 800	900
15	10	400 – 600	1000
16	10	200 – 400	900
4	15	400 – 600	1100
9	15	400 – 600	900
11	15	200 – 400	1000
14	15	600 – 800	1000

CHAPTER FOUR

4. RESULT AND DISCUSSION

4.1. Chemical analysis

Results of the chemical analysis of the raw clay soil materials were presented in Table 4.1.

Table 4. 1: - Chemical Analysis of Red Clay Soil (Soil pH=5.8)

No.	Element oxide	Percentage of elements in raw clay
1	Fe ₂ O ₃	12.4
2	SiO ₂	3.27
3	Al ₂ O ₃	2.72
4	K ₂ O	3.02
5	Na ₂ O	5.55
6	MgO	3.01
7	CaO	3.34
8	Others insoluble and organic materials	66.7

The precipitation gravimetric analysis obtained from the above table clearly shows that the soil is clay soil and it can be used for the manufacturing of porous ceramic filter pots. The presence of SiO₂ and Al₂O₃ indicates the existence of kaoline, the major mineral in clay soil. The existence of Fe₂O₃, SiO₂ and Al₂O₃ at this range is important to stick molecules of the soil together during firing. The result from the analysis found to be nearly the same with the characterization study of clay soil by (Akbar et al., 2017). It is also observed that the amount of Fe₂O₃ is the major element found from the chemical analysis which is responsible for the redish color of the raw clay soil.

4.2. Ash content analysis of sawdust

Result of ash content analysis of sawdust material is presented below on the table 4.2

Table 4. 2: - Ash content analysis of sawdust at different sizes

Sample No.	Particle size (µm)	Sawdust weight (g)	Ash weight (g)	Ash weight (%)
1	200 - 400	2	0.20	10%
2	400 - 600	2	0.16	8%
3	600 - 800	2	0.10	5%

From the table, it is observed that when the particle size decreases the ash content increase due to the fact that the smaller particle sizes are less coarse, compact easily leading to incomplete combustion due to small numbers of pore spaces which affects the porosity of the AgNP impregnated porous ceramic water filter pot. Coarse particle sizes have greater number of pore spaces allowing oxygen to flow easily within the sample. However, for the medium sizes the compaction was moderate thus there was available space for the oxygen to flow and combustion was near complete. Lastly, the larger particle which were the most difficult to compact were more loosely bond. Hence, allowing adequate flow of oxygen and so combustion was complete resulting in less ash content which when the particle size is larger, the ash content decreases and the porosity also increases (Plappally et al., 2011).

4.3. UV-Vis Spectroscopy analysis to determine the formation of AgNP

From the first figure (figure 4.1 (A) and (B)) we can observe that the change of color to pale yellow which indicates the formation of silver nanoparticles.

Further analysis using UV-Visible Spectro photometer analysis also confirmed as shown on Figures 4.2 and 4.3 below, the obtained UV-Visible absorption spectra of pure NaBH₄ solution and NaBH₄:AgNO₃ solution samples. As can be seen in the Figure 4.3 the pure does not show any appreciable absorption peak in the wavelength region 200 nm to 800 nm, however a broad absorption band centered at 390 nm was observed for NaBH₄: AgNO₃ solution samples (Figure 4.2). This absorption band is called a localized surface plasmon resonance (SPR). Other researchers like (De Leersnyder et al., 2019) also observed the SPR peak in the same region for spherical silver nanoparticles produced using sodium borohydride as a reduction agent. Thus, the observed SPR in NaBH₄:AgNO₃ samples reveals that the reduction of silver ions (Ag⁺) to the metallic silver nanoparticles (Ag⁰) in the present system.

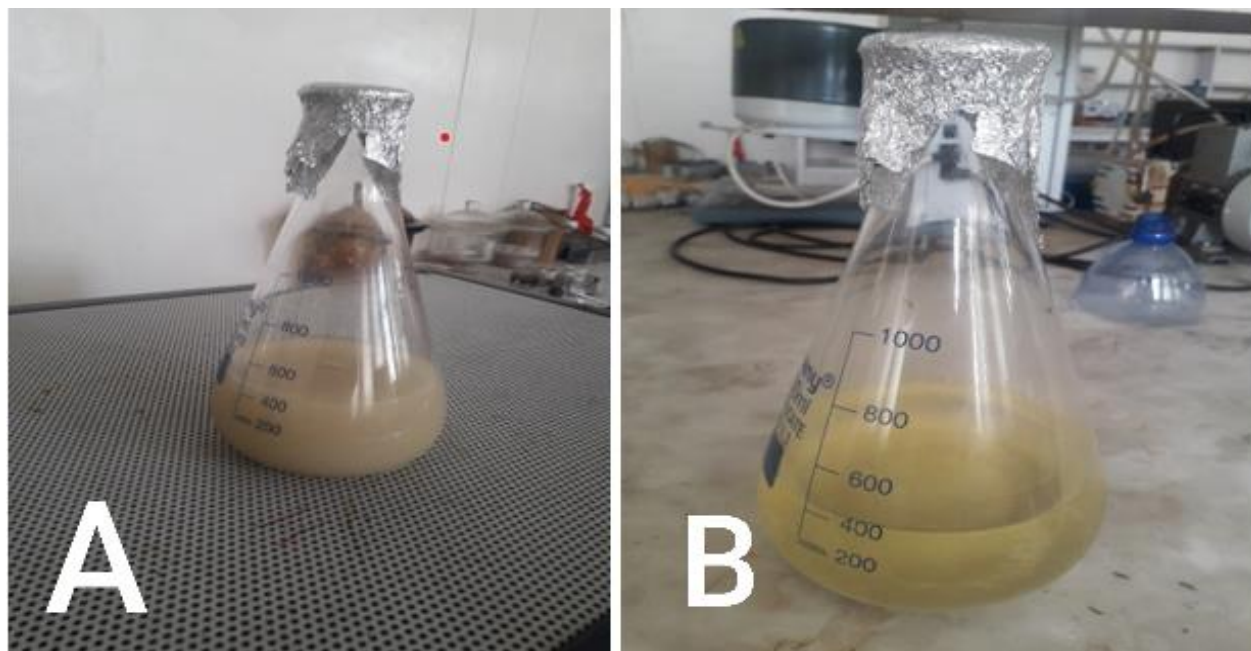


Figure 4. 1: - The color change of the reaction of silver nitrate and sodium borohydride to pale yellow in the presence of SDS

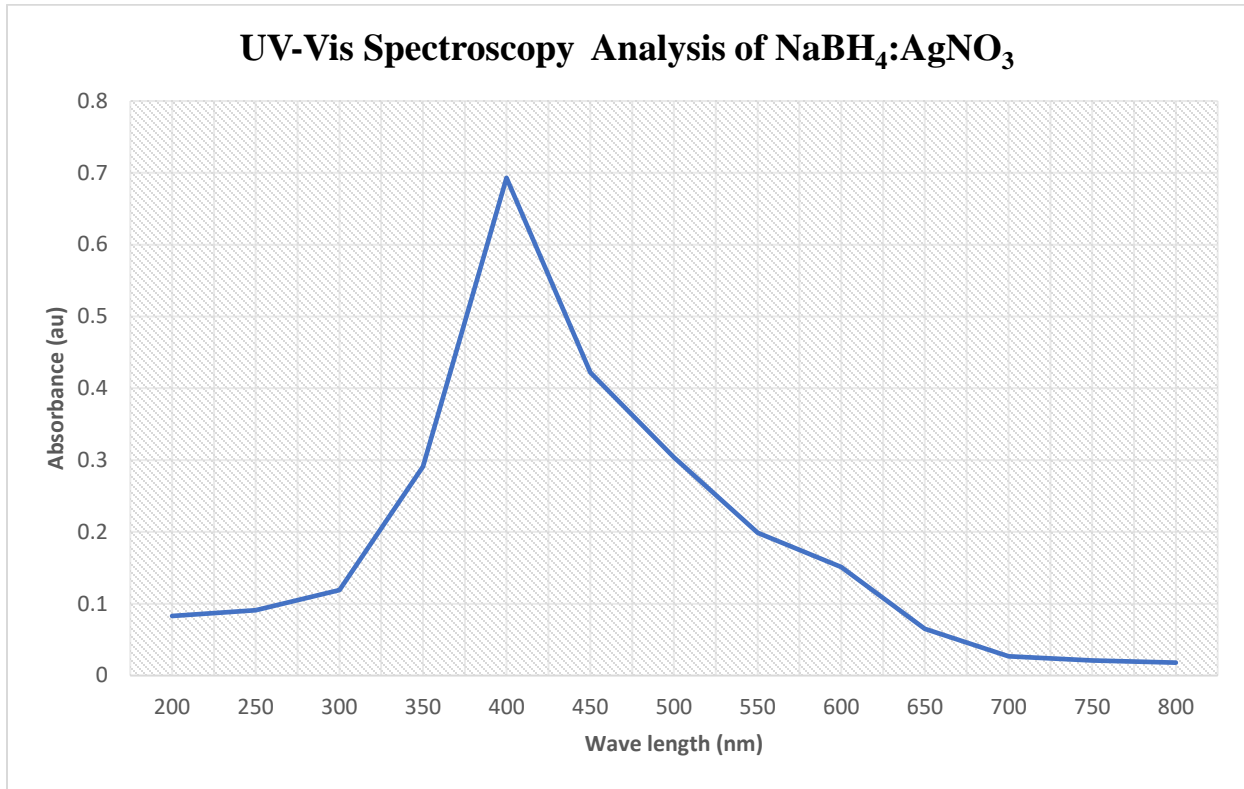


Figure 4. 2: - Absorption spectra of NaBH₄:AgNO₃ solution

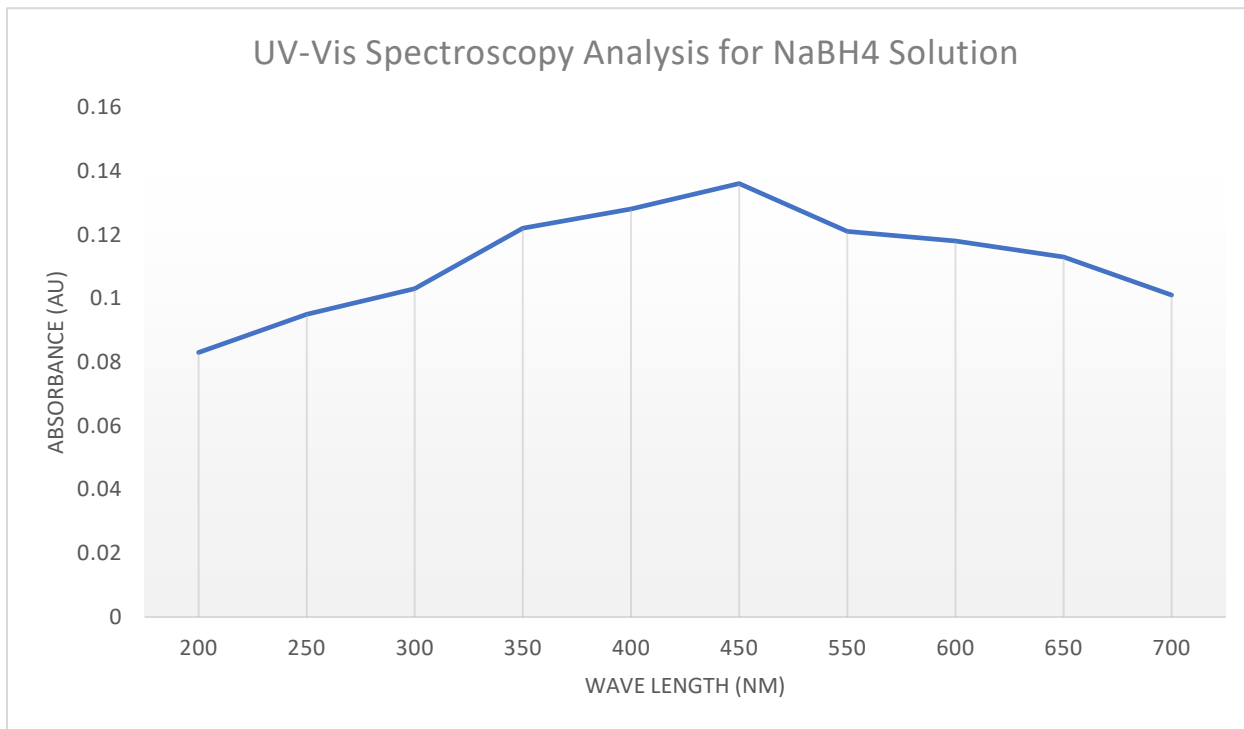


Figure 4. 3: -Absorption spectra of NaBH₄ solution

4.4. Scanning Electron Microscope (SEM)

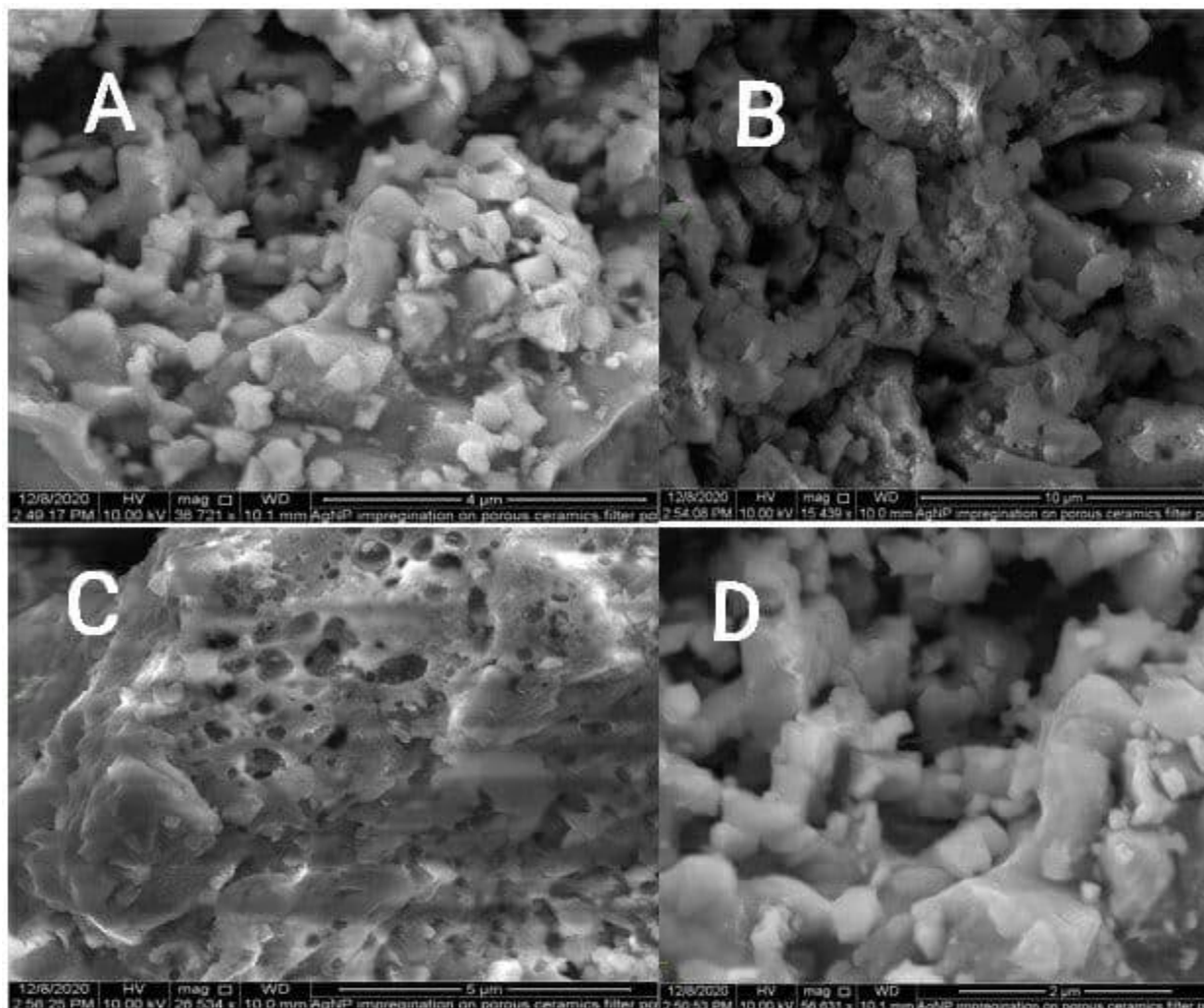


Figure 4. 4: - SEM image AgNP deposition on the ceramic water filter pot at different magnification (A = 4 μm, B = 10 μm, C = 5 μm, D = 2 μm)

From the above SEM image it can be concluded that the ceramic water filter was porous and silver nanoparticles are impregnated on the surfaces and inside the pores of the porous ceramic water filter pots. The morphology of the SEM image shows the existence of silver nanoparticles. The white spiral shapes indicate the existence of silver nanoparticles. According to Geoprincy et al., study on the combined effect of silver nanoparticles and antibiotics, it was found nearly the same SEM images of AgNP's impregnation over the porous ceramic water filters disks that supports the result found here (Geoprincy et al., 2011).

4.5. Porosity Test/Determination

Table 4. 3: - Percentage porosity of manufactured AgNP impregnated ceramic pot filter samples.

Runs	Actual factors			Calculated % of porosity
	sawdust amount used (%)	Particle size of sawdust (μm)	Furnace firing Temperature ($^{\circ}\text{C}$)	
2	5	200 – 400	1000	26.74730
5	5	400 - 600	900	28.88218
10	5	400 - 600	1100	27.38632
17	5	600 – 800	1000	29.33265
1	10	200 – 400	1100	30.38961
3	10	400 - 600	1000	30.66022
6	10	600 – 800	1100	30.89136
7	10	400 - 600	1000	30.88132
8	10	400 - 600	1000	31.04035
12	10	400 - 600	1000	31.03992
13	10	600 – 800	900	31.58365
15	10	400 - 600	1000	31.04652
16	10	200 – 400	900	30.22871
4	15	400 - 600	1100	36.19551
9	15	400 - 600	900	36.28460
11	15	200 – 400	1000	34.88470
14	15	600 – 800	1000	39.99153

As demonstrated on the above table, the porosity of the AgNP impregnated porous ceramic water filter pots estimated to be from 26.7473% to 39.99153%. It indicates that as the mass percentage and particle size of sawdust increases the porosity also increases. As we saw from runs 3, 8, 13 and 15 which the test is done under the same factors clearly shows that there is no difference in filtration rate. Particle size and mass percentage of sawdust affected the process highly whereas the effect of furnace firing temperature is low. Filtration rate and bacterial removal efficiency also related on the pore size of the ceramic water filter pots. As the pore size increases the amount of filtrate recovered also increases where as the bacterial removal efficiency decrease because as the pore size increases the bacteria easily escape to the filtrate.

Table 4. 4: - Response 1: Filtration rate

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	5.09	9	0.5660	2820.10	< 0.0001	significant
A-Percentage of Sawdust	2.84	1	2.84	14169.96	< 0.0001	
B-Sawdust Screen Size	2.11	1	2.11	10519.99	< 0.0001	
C-Furnace Temperature	0.0840	1	0.0840	418.75	< 0.0001	
AB	0.0169	1	0.0169	84.20	< 0.0001	
AC	0.0002	1	0.0002	1.12	0.3249	
BC	0.0030	1	0.0030	15.07	0.0060	
A ²	0.0121	1	0.0121	60.04	0.0001	
B ²	0.0099	1	0.0099	49.34	0.0002	
C ²	0.0089	1	0.0089	44.39	0.0003	
Residual	0.0014	7	0.0002			
Lack of Fit	0.0003	3	0.0001	0.4012	0.7605	not significant
Pure Error	0.0011	4	0.0003			
Cor Total	5.10	16				

The model F-value of 2820.10 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, BC, A², B², C² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the model. The lack of fit F-value of 0.40 implies the lack of fit is not significant relative to the pure error. There is a 76.05% chance that a lack of fit F-value this large could occur due to noise. Non-significant lack of fit is good since we want the model to fit.

Table 4. 5: - Filtration rate fit Statistics

Std. Dev.	0.0142	R ²	0.9997
Mean	1.87	Adjusted R ²	0.9994
C.V. %	0.7567	Predicted R ²	0.9986
		Adeq Precision	204.3094

The predicted R² of 0.9986 is in reasonable agreement with the adjusted R² of 0.9994; i.e. the difference is less than 0.2. Adequacy precision measures the signal to noise ratio. A ratio greater

than 4 is desirable. The ratio of 204.309 indicates an adequate signal. This model can be used to navigate the design space.

Table 4. 6: - Coefficients in Terms of Coded Factors

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	1.94	1	0.0063	1.93	1.96	
A-Percentage of Sawdust	0.5962	1	0.0050	0.5844	0.6081	1.0000
B-Sawdust Screen Size	0.5138	1	0.0050	0.5019	0.5256	1.0000
C-Furnace Temperature	-0.1025	1	0.0050	-0.1143	-0.0907	1.0000
AB	0.0650	1	0.0071	0.0482	0.0818	1.0000
AC	-0.0075	1	0.0071	-0.0243	0.0093	1.0000
BC	0.0275	1	0.0071	0.0107	0.0443	1.0000
A ²	-0.0535	1	0.0069	-0.0698	-0.0372	1.01
B ²	-0.0485	1	0.0069	-0.0648	-0.0322	1.01
C ²	-0.0460	1	0.0069	-0.0623	-0.0297	1.01

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multicollinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

Table 4. 7: - Response 2: Log reduction of E. coli

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	62.34	9	6.93	1258.93	< 0.0001	significant
A-Percentage of Sawdust	30.29	1	30.29	5505.00	< 0.0001	
B-Sawdust Screen Size	26.77	1	26.77	4864.86	< 0.0001	
C-Furnace Temperature	0.3015	1	0.3015	54.80	0.0001	
AB	3.05	1	3.05	553.46	< 0.0001	
AC	0.6068	1	0.6068	110.30	< 0.0001	
BC	0.4376	1	0.4376	79.53	< 0.0001	
A ²	0.0565	1	0.0565	10.28	0.0149	
B ²	0.7204	1	0.7204	130.93	< 0.0001	
C ²	0.0598	1	0.0598	10.86	0.0132	
Residual	0.0385	7	0.0055			
Lack of Fit	0.0174	3	0.0058	1.10	0.4454	not significant
Pure Error	0.0211	4	0.0053			
Cor Total	62.38	16				

The model F-value of 1258.93 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, AC, BC, A², B², C² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The lack of fit F-value of 1.10 implies the lack of fit is not significant relative to the pure error. There is a 44.54% chance that a lack of fit F-value this large could occur due to noise. Non-significant lack of fit is good.

Table 4. 8: - E. coli log reduction fit Statistics

Std. Dev.	0.0742	R ²	0.9994
Mean	2.76	Adjusted R ²	0.9986
C.V. %	2.69	Predicted R ²	0.9950
		Adeq Precision	132.7100

The Predicted R^2 of 0.9950 is in reasonable agreement with the adjusted R^2 of 0.9986; i.e. the difference is less than 0.2. Adequacy precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 132.710 indicates an adequate signal. This model can be used to navigate the design space.

Table 4. 9: - Coefficients in Terms of Coded Factors

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	2.45	1	0.0332	2.38	2.53	
A-Percentage of Sawdust	-1.95	1	0.0262	-2.01	-1.88	1.0000
B-Sawdust Screen Size	-1.83	1	0.0262	-1.89	-1.77	1.0000
C-Furnace Temperature	0.1941	1	0.0262	0.1321	0.2561	1.0000
AB	0.8725	1	0.0371	0.7848	0.9602	1.0000
AC	0.3895	1	0.0371	0.3018	0.4772	1.0000
BC	-0.3307	1	0.0371	-0.4184	-0.2431	1.0000
A ²	0.1159	1	0.0361	0.0304	0.2014	1.01
B ²	0.4136	1	0.0361	0.3281	0.4991	1.01
C ²	0.1191	1	0.0361	0.0336	0.2046	1.01

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multicollinearity, the higher the VIF the more severe the correlation of factors.

4.7. Effect of porosity on flow rate and the permeability of AgNP impregnated Porous ceramic water filter pot

The most porous of the filters studied is, fabricated using 15% sawdust and with highest size of sawdust screen size of in the range of 600 μ m to 800 μ m, exhibited the fastest discharge of water of 3.02 L/hr. The filter with the lowest flow rate (0.79 L/hr.) in the study was the ceramic pot made up of with 5% sawdust and sawdust particle size of in the range from 200 μ m to 400 μ m. Therefore, the rate of water flow by AgNP impregnated CWFP increases with the percentage of sawdust and particle size of sawdust used in making the filter. The average flow rate obtained from filter pot of

run/code 2, 3, 12, 14 and 17 at 10% of sawdust and screen size from 400 to 600, of AgNP impregnated CWFP was 1.95 L/hr and on which the average all runs tested.

4.8. Fabricated porous ceramic pots Flow rate test

The flowrate test is done after soaking ceramic water filter pots with distilled water for twenty-four hours to have a consistent flow through the pores. Distilled water was used to minimize the error that would happen during bacterial removal tests. The result showed that all the three factors altered the flowrate at different intensity. The effect of each three factors and their combination effect discussed below.

4.8.1. Effects of percentage of sawdust on filtration

The performance of CWFP can be significantly improved by the use of different amount of burnout materials which increase flow rate by creating a network of pores and the use of bactericidal compounds for destruction of pathogens. As shown from the figure 4.3 below the porosity of the ceramic water filter pot increased with an increase in the amount of sawdust. The flow rate through the filters was found to vary with increase in proportion of sawdust. Figure 4.3 shows that the flow rate varied from 0.79 liters/hr for the filter with 5% of mass of sawdust to 3.02 l/hr for filter with 15% mass of sawdust. The most porous of the ceramic water filter pot studied (15% mass of water filter), exhibited the fastest discharge, followed by the ceramic water filter pot made by 10% mass of sawdust. The slowest discharge rates were associated with ceramic water filter pot made by the 5% mass fraction of sawdust. Therefore, the rate of water discharge by CWFP increases with porosity, or in other words with the mass fraction of sawdust used in making the filter.

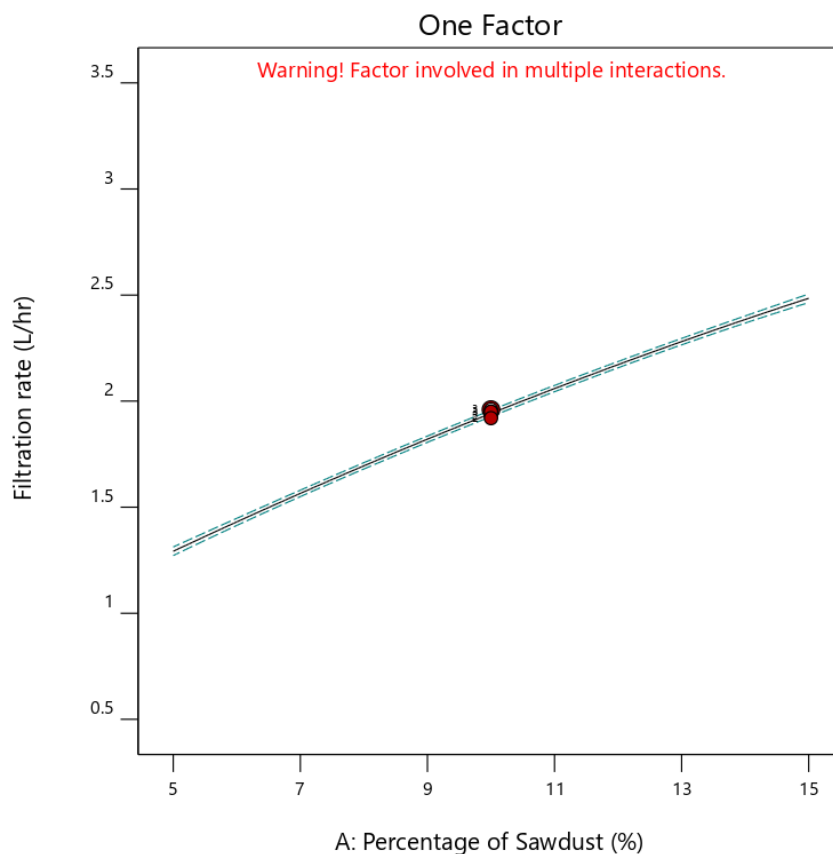


Figure 4.5: - effect of mass percentage of sawdust on the filtration rate of CWFP

4.8.2. Effect of screen size/particle size of Sawdust on filtration

The other major effect on filtration rate of AgNP impregnated CWFP studied in this research was the screen size of sawdust. It can be clearly seen that the screen size of the sawdust had effects on the porosity of CWFP's by looking carefully the variation of ash content due to the screen size of the sawdust. The ash content decreases with increasing the screen size of the sawdust that leaves large pore sizes on fired CWFP's and filtration rate altered with in the variation of pore sizes.

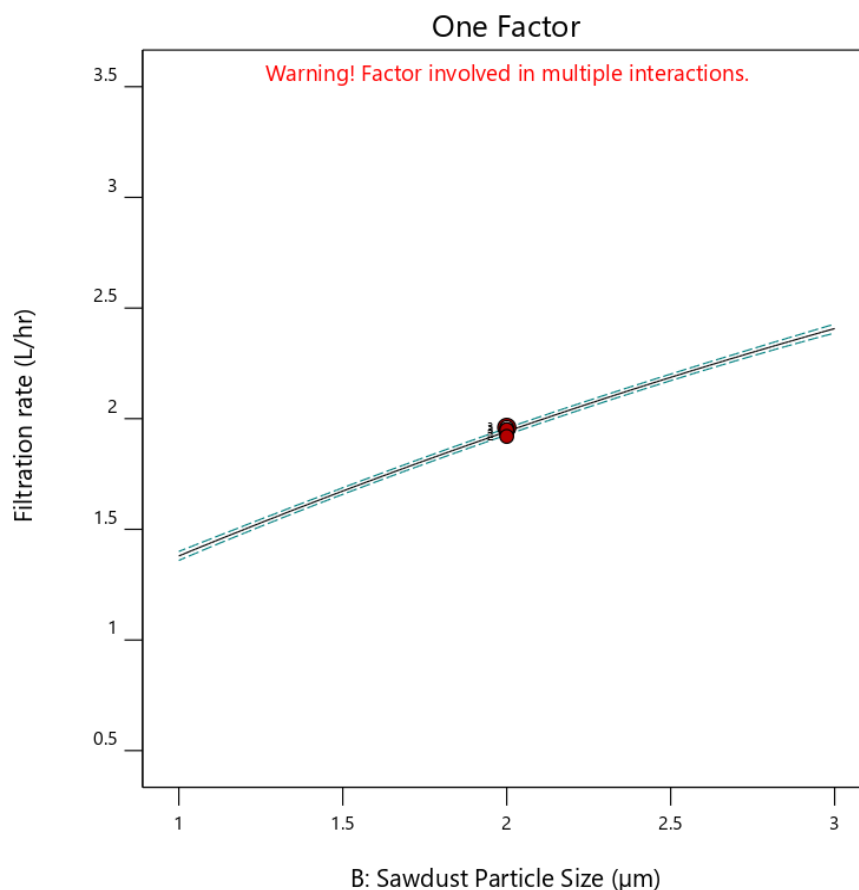


Figure 4. 6: - effect of screen size/particle size of sawdust on the filtration rate of CWFP

As seen from the above figure (fig) the filtration rate increases with increasing the screen size of the burnout material sawdust. The maximum amount of filtrate was recovered at 3 i.e. from the screen size of sawdust in between 600 to 800 µm.

4.8.3. Effect of furnace firing temperature

Furnace firing temperature had little effect on the filtration rate of CWFP's compared to the mass percentage amount of sawdust and screen size of the sawdust. Increase in furnace firing temperature can make particles of clay soil to stick more together and affect the formation of pores.

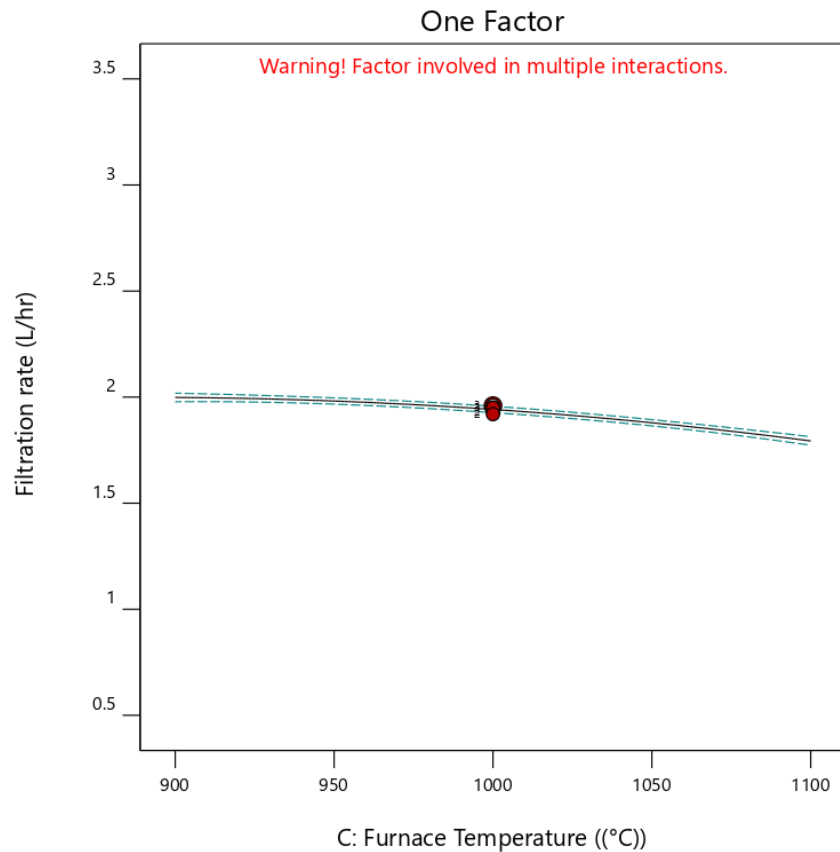


Figure 4. 7: - Effect of firing furnace temperature on the filtration rate of CWF

The analysis from the above figure shows that increase in firing temperature lead for the decrease in the filtration rate of the porous ceramic water filter pots. Run 6 and run 16 with the same mass percentage and screen size of sawdust clearly showed that the impact of firing furnace temperature with different firing furnace temperatures at 900 °C and 1100 °C. The amount of recovered from run 16 which was fired at a temperature of 900 °C 2.55 L/hr and run 6 with a firing temperature of 1100 °C is 2.32 L/hr.

4.8.4. Interaction effect of multiple factors

4.8.4.1. Effect of mass percentage and screen size of sawdust on filtration rate

The figure below indicates that, the interaction between effect of mass percentage and screen size of sawdust was significant and affect the response. From the ANOVA table, the p-value of mass percentage and screen size of sawdust was less than <0.0001 . This means the p-value of the interaction is less than the p-value of the model design. Generally, there was combined effect of mass percentage and screen size of sawdust observed on the filtration rate. So, the filtration rate

was dependent of influence of mass percentage of sawdust and screen size of sawdust at the same time. The proportionate increment of mass percentage of sawdust and screen size of sawdust was significantly affecting the filtration rate. When mass percentage of sawdust goes from 5 to 15% and screen size of sawdust goes from 200 μm to 800 μm it gives high filtration rate.

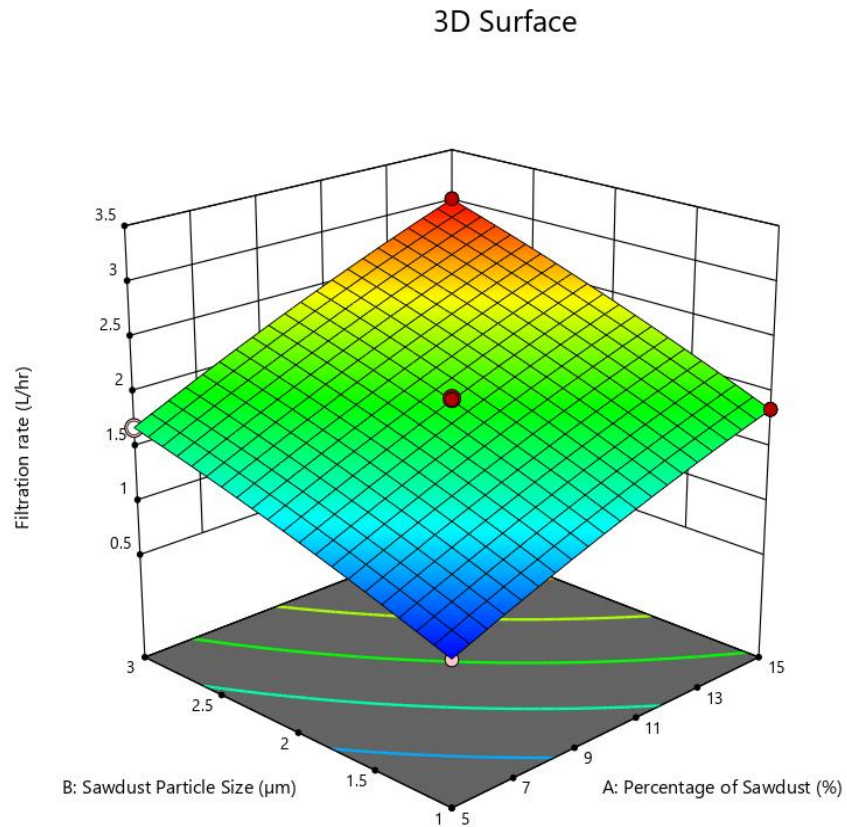


Figure 4. 8: - Interaction effect of mass percentage and screen size of sawdust

4.8.4.2. *Effect of furnace firing temperature and mass percentage of sawdust*

The figure below indicates that, the interaction between temperature and mass percentage of sawdust. The filtration rate is higher when mass percentage of sawdust gradually increased from 5 to 15% and furnace firing temperature decreased from 1100 to 900 $^{\circ}\text{C}$. The result found at these both operational conditions were 3.2 L/hr. The major observable issue here is the interaction of these factors was counter currently; i.e. when mass percentage of sawdust increases and furnace firing temperature decreases, the filtration rate also increased.

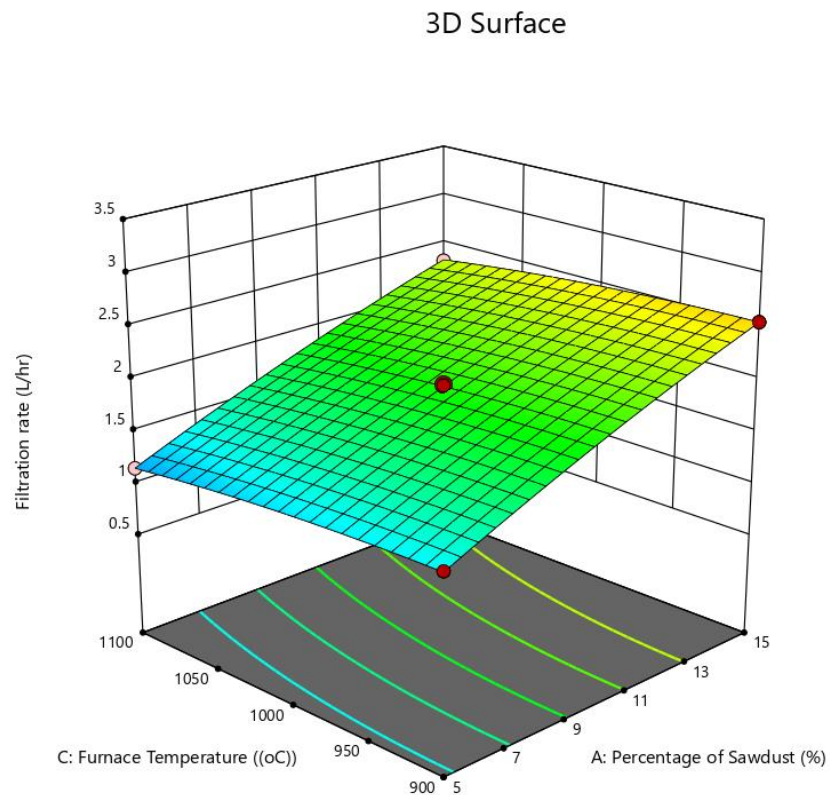


Figure 4. 9: - Interaction effect of furnace firing temperature and mass percentage of sawdust

4.8.4.3. *Effect of screen size of sawdust and furnace firing temperature*

The figure below indicates that, the interaction between effect of screen size of sawdust and furnace firing temperature affect the response. The combined effect of screen size of sawdust and furnace firing temperature observed on the filtration rate. The interaction of these factors was counter currently; i.e. when screen size of sawdust increases and furnace firing temperature decreases. As screen size of sawdust gradually increased from 200 μm to 800 μm and furnace firing temperature decreased from 1100 $^{\circ}\text{C}$ to 900 $^{\circ}\text{C}$ the filtration rate was 3.2 L/hr.

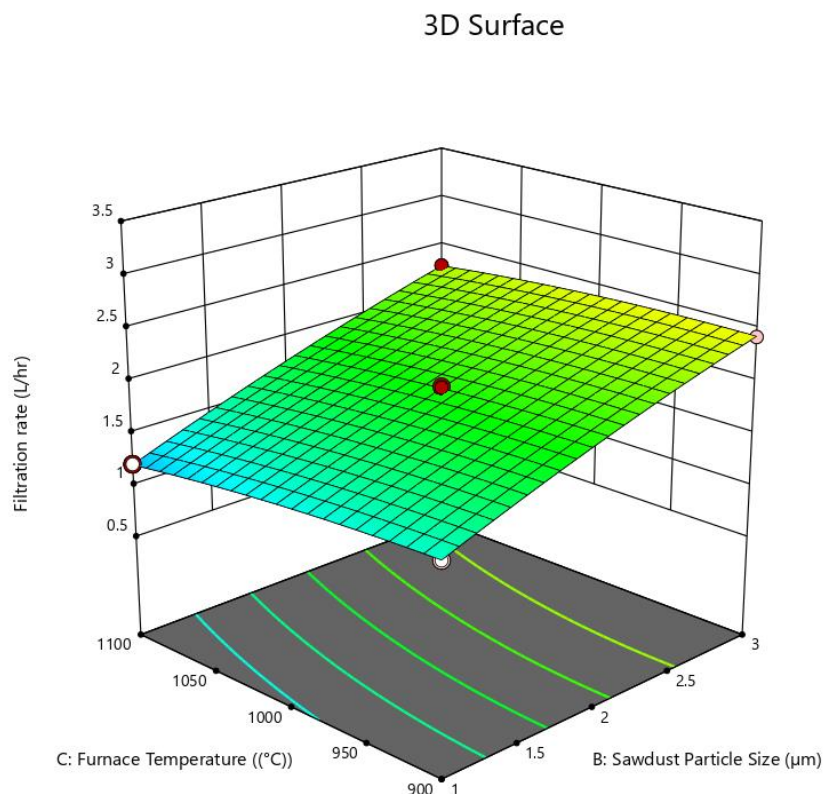


Figure 4. 10: - Interaction effect of screen size of sawdust and furnace firing temperature

4.9. Bacterial removal efficacy of AgNP impregnated porous ceramic Pot

The initial concentrations of *E. coli* and total coliform in peptone buffered water which was used for microbial removal test were estimated to be 3.82×10^8 CFU/100 ml. *E. coli* removal efficiency of the AgNP impregnated ceramic water filter pots were ranged from 85.365 to 99.99%. Also, the result of this study showed that the microbial removal efficiency of the ceramic filters, at uniform application of AgNP, increases with increase in the percentage composition of clay, firing temperature and decrease in screen size of sawdust. The least removal efficiency registered are 85.365% and 87.804% for total *E. coli* coliform for ceramic water filter run/(code) 13 and 16. This is most likely obtained due to high porosity of the filter element because of the high percentage composition and high in screen size of burn out material beside AgNP which will kill some of *E. coli* coliforms. AgNP impregnated Ceramic water filters with relatively low porosity have good efficiency in removing microbial from bacterially contaminated water sources in either by filtering or increasing the contact time with AgNP that will kill the bacteria. When microorganism pass

through the pores of ceramic filters, there might be strong suffocation on their path due to the tortuosity of the path and at the same time they compete for feeding which reduce the number of microbials after filtration (J. Brown & Sobsey, 2009). The interaction and effect of factors on E. coli removal efficiency is discussed below with log reduction model graphs found from ANOVA analysis.

4.9.1. Effect of percentage of sawdust

Bacterial removal efficiency decreased with increase in percentage of sawdust. Increase in mass percentage of sawdust increases the porosity of the AgNP impregnated ceramic water filter pot so that the filtration rate increases and bacterial removal efficacy decreases. The log reduction of E. coli by the AgNP impregnated ceramic water filter pot graph presented below on figure 4.7.

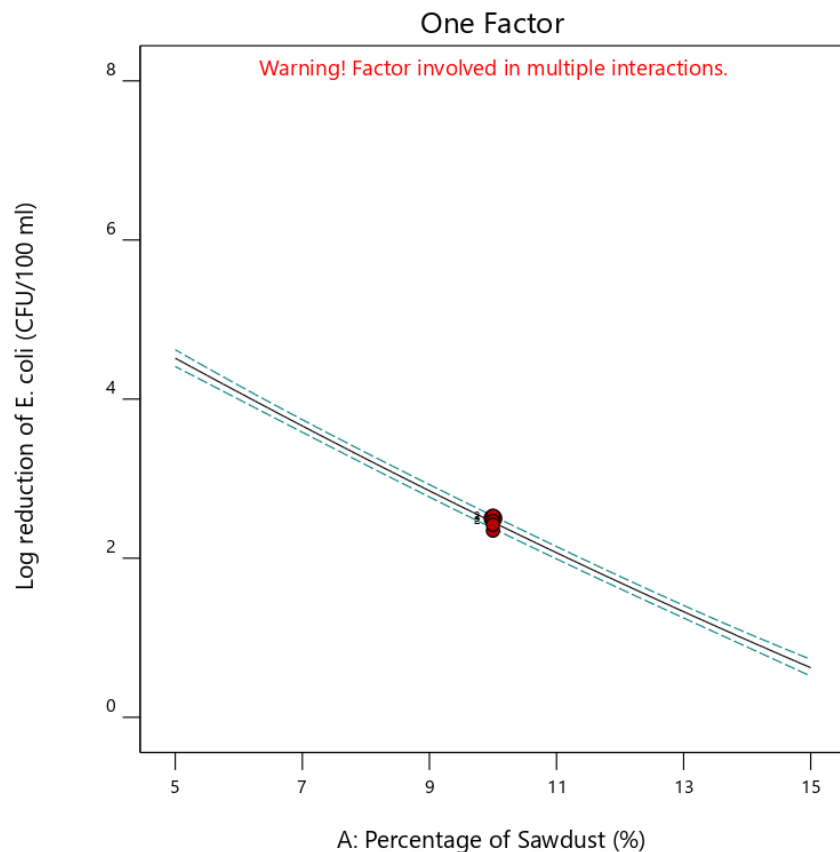


Figure 4. 11: Effect of mass percentage of sawdust on bacterial removal efficacy

4.9.2. Effect of screen size

The screen size of sawdust is also one of the factors in bacterial removal efficiency of AgNP impregnated porous water filter ceramic pot. Increase in screen size will decrease the amount of ash content of the burnout material which will increase the porosity of the ceramic water filter pot.

It can be seen from the below log reduction graph that when the screen size/particle size of sawdust increase from 200 μm to 800 μm , the log reduction of E. coli decreases. It is because of increase in porosity of the ceramic water filter pot which decreases the contact time of silver nanoparticles.

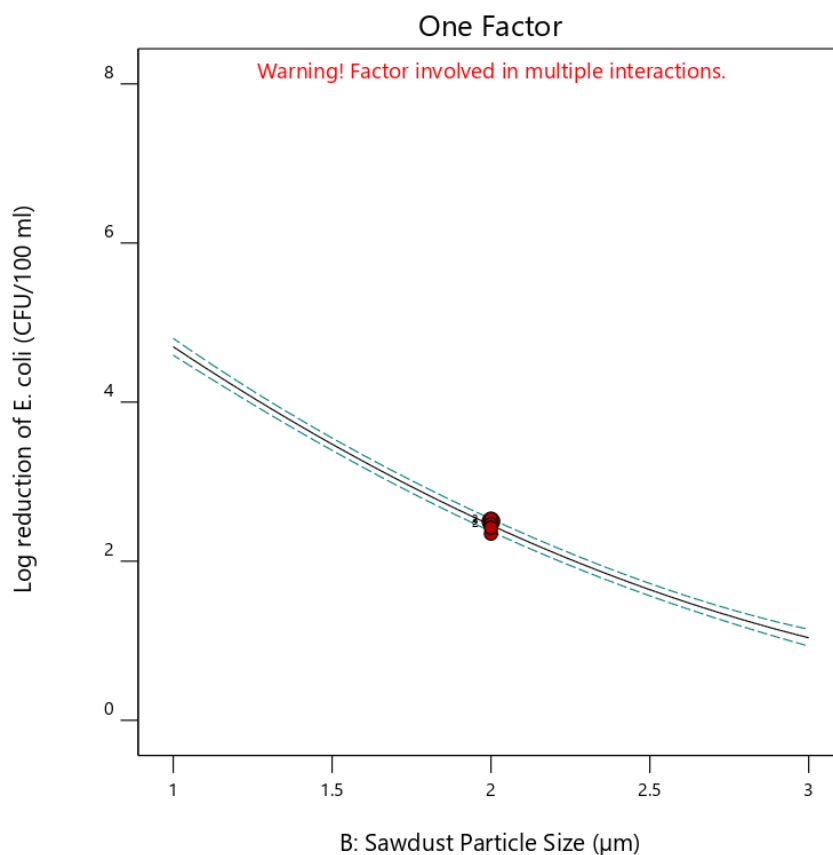


Figure 4. 12: Effect of screen size of sawdust on bacterial removal efficacy

4.9.3. Effect of Furnace temperature

The firing furnace temperature has little effect on the log reduction of E. coli compared to screen size of sawdust and mass percentage of sawdust. When the firing furnace temperature increases from 900 to 1100 $^{\circ}\text{C}$, the porosity of the AgNP impregnated ceramic water filter pot decreases due to the sticking of the particles of raw materials that make the CWFP. Decrease in porosity increases

the contact time of the pathogen like E. coli with silver nanoparticles so that the silver nanoparticles interact with E. coli membranes which causes the inactivation or death of E. coli.

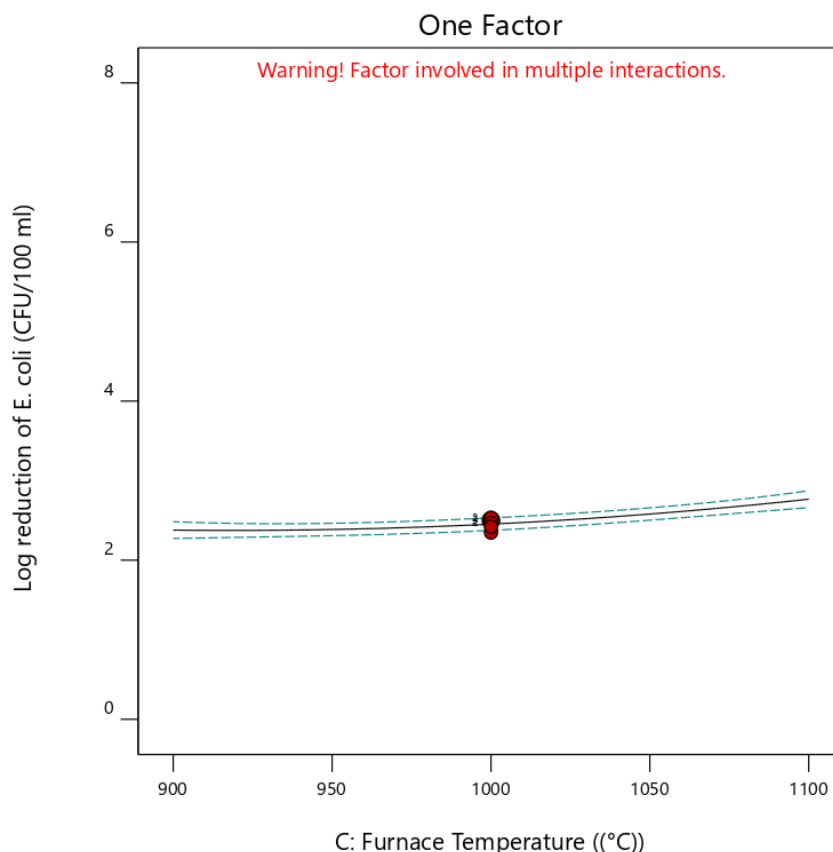


Figure 4. 13: - Effect of furnace temperature on bacterial removal efficacy

As we saw from the above graph from the ANVOVA analysis, the log reduction of E. coli increased as the firing furnace temperature increased from 900 to 1100 °C.

4.9.4. Interaction Effect of multiple factors on Log reduction of E. coli

It can be seen from the ANOVA analysis that both factors alter the E. coli removal efficacy of the AgNP impregnated porous ceramic water filter pots. According to the study of this research it is observed that in the case of bacterial removal efficacy, increase in mass percentage and screen size of the sawdust affects the response negatively but in the case of filtration rate it alters positively whereas the firing temperature had the reverse impact.

4.9.4.1. The effect of mass percentage and screen size of sawdust

As it has been seen from the figure below which indicates that both increase in mass percentage and screen size of sawdust alters the removal efficacy/log reduction of E. coli negatively.

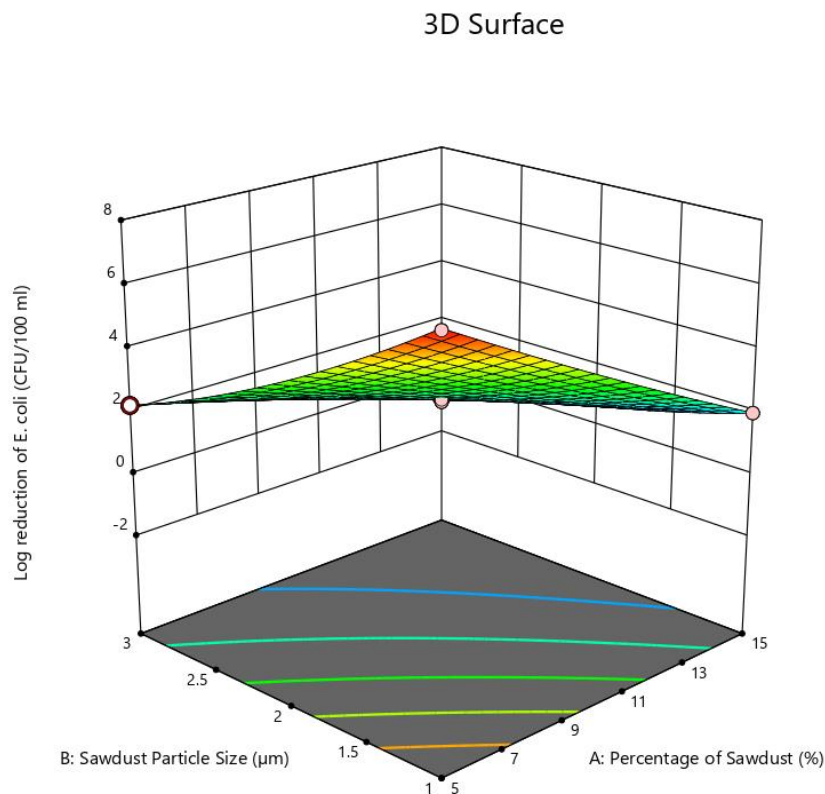


Figure 4. 14: - Interaction effect of mass percentage and screen size of sawdust

It was observed that from the single factor interaction, increase in mass fraction and screen size of sawdust was decreasing the removal efficacy of E. coli. The interaction of both mass fraction and screen size of sawdust also affects more the removal efficacy E. coli. The minimum E. coli removal efficiency was found at maximum percentage amount of sawdust (15%) and maximum screen size of sawdust (800µm).

4.9.4.2. Effect of the mass percentage of sawdust and furnace firing temperature on log reduction of E. coli

The figure below indicates that both mass percentage of sawdust and furnace firing temperature affects E. coli removal efficacy of the AgNP impregnated porous ceramic water filter pots. It is clearly seen from the figure that increase in mass percentage of sawdust from 5% to 15% decreases

bacterial removal efficacy while increase in furnace firing temperature from 900 °C to 1100 °C increased E. coli removal efficiency of the AgNP porous ceramic water filter pot. It is observed that increase in mass percentage of sawdust increases the porosity of the CWFP which allows E. coli to pass through on the pores easily by decreasing the contact time with silver nano particles. Whereas increase in furnace firing temperature decrease the porosity of CWFP due to the agglomeration and sticking property of particles that made up of the ceramic water filter pot. Decrease in porosity also increases the removal efficacy of E. coli by increasing the efficiency of filtration and contact time with silver nanoparticles which kills the E. coli.

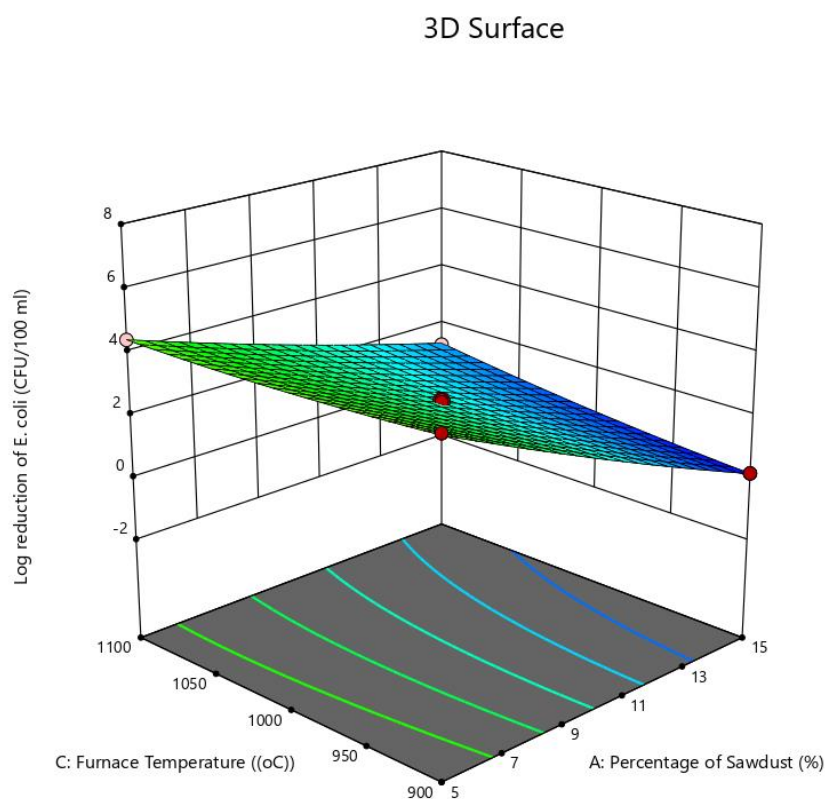


Figure 4. 15: - Combination effect of sawdust mass percentage and furnace firing temperature

4.9.4.3. *Effect of the screen size/particle size of sawdust and furnace firing temperature on log reduction of E. coli*

The combined effect of screen size of the sawdust and furnace firing temperature observed from the figures below. When the screen size of sawdust increase from 200 µm to 800 µm then the

bacterial removal efficiency decreases where as in the case of furnace firing temperature it is a vise versa. Therefore, the two factors have a counter current effect on the E. coli removal response.

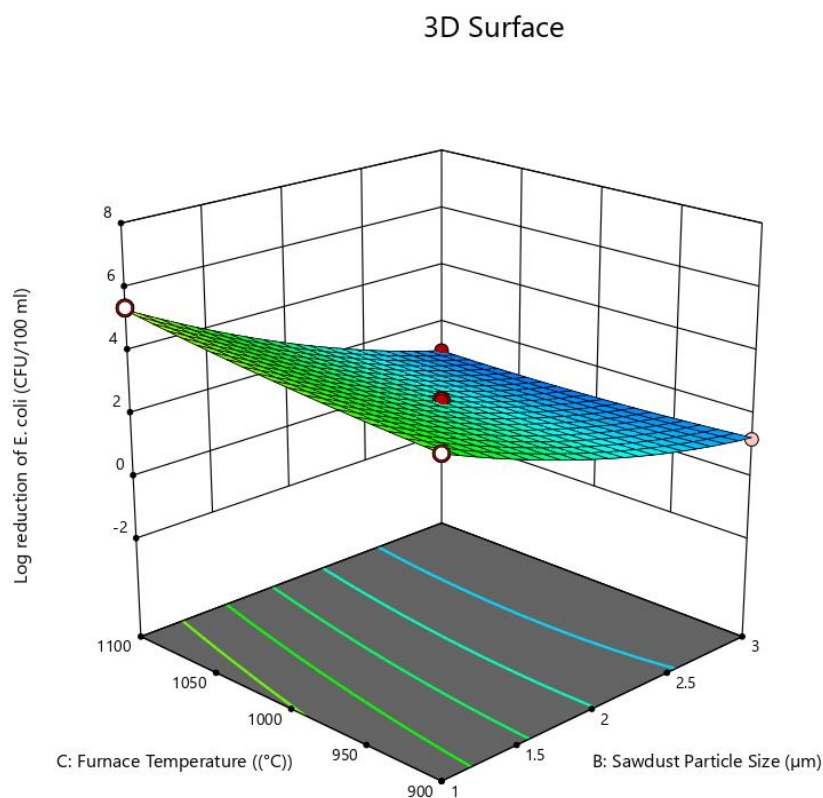


Figure 4. 16: - Combination effect of sawdust screen size and furnace firing temperature

4.10. Swamp surface Microbial removal efficiency

The initial concentrations of E. coli and total coliform of sample taken from Kebena river water which was used for microbial removal test were found to be 107 CFU/100 ml. Four samples with two of them have same variables (run/code 3 and 12) and two others having different variables (run/code 13 and 15) was selected and were tested to check the impact of factors and the efficiency of the AgNP impregnated ceramic filters to remove total coli form and E. coli form bacteria from Kebena river water. The total removal efficiency ranges from 98.13% to 99.999% . Also, the result of this study showed that the microbial removal efficiency of the AgNP impregnated ceramic water filters increase with increase in the percentage composition of clay and firing temperature which the furnance firing temperature cause decrease in size of porosity as the firing temperature increases that will lead the sticking of molocules together. The least removal efficiency registered

98.3% for total coli form and *E. coli* for ceramic water filter run/code 13. This is most probably obtained due to high porosity of the filter element because of the high percentage composition of burn out. Ceramic water filters with relatively low porosity have good efficiency in removing microbial from bacterially contaminated water sources (Clasen, 2008). When microorganism pass through the pores of ceramic filters, there might be strong suffocation on their path due to the tortuosity of the path and at the same time they interact with AgNP's which reduce the number of viable microbials after filtration. The result of this study showed that most of the developed AgNP impregnated ceramic water filter pots for this work were found to be effective in removing microorganisms from contaminated water as compared with WHO guidelines (Onda et al., 2012a; WHO, 2019).

4.11. Optimization of the process variables

The above results shown that the three variables and their interaction affected the responses of the process. The next step is to find the optimum point of the maximum bacterial (*E. coli*) removal efficiency and the maximum filtration rate of AgNP impregnated ceramic water filter pot at the optimum points of process variables with suggested ANOVA regression model. Therefore, from the regression model, it is shown that at the percentage mass of sawdust 8.1%, particle size of sawdust in between 400 to 600 μ m and at furnace firing temperature of 900 °C, the average rate of water collected from CWFP's and average bacterial (*E. coli*) removal efficiency is respectively determined to be 1.652 L/hr and 99.96% (with an average log reduction of 3.73). This result had a better output comparing with Sandeep Gupta et al. research (S. Gupta et al., 2018) which had an average filtration rate result of 1.32 L/hr and 99% removal efficiency of *E. coli*.

To validate the optimum condition found, triplicate experiments were conducted using the optimum process variables and the results obtained are nearly the same with the data obtained from optimization analysis.

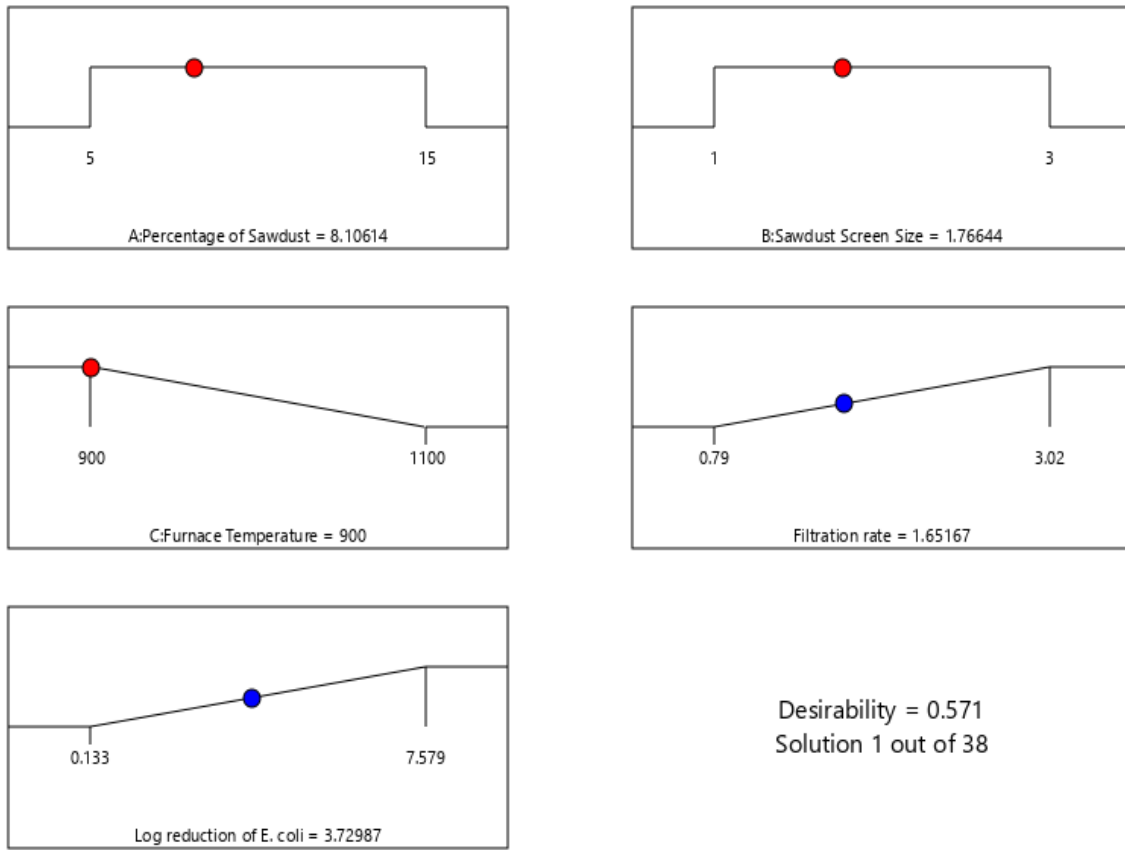


Figure 4. 17: - The analysis of optimum point of factors

CHAPTER FIVE

5. CONCLUSION AND RECOMMENDATIONS

5.1. Conclusion

The implications of the results in this thesis work suggest that AgNP impregnated ceramic water filter pots with different porosities can be used to filter out most of the bacterial pathogens in water. With *E. coli* removal efficacy of approximately from 85.6 to 99.999%, the use of AgNP impregnated ceramic water filters can impact significantly to the removal of microbial pathogens from drinking water in the developing world including in our country which most of the people lives scattered in rural areas which is hard to supply water through pipes and where about 1.8 million people die every year all over the world due to diarrhea from the effects of consuming contaminated water. The porosity, permeability and overall flow rates increase with increasing percentage mass ratio and screen size of sawdust whereas it decreases with increasing firing furnace temperature. An average flow rate of 1.652 L/hr was obtained from the AgNP impregnated porous ceramic water filter pots with a sawdust percentage mass ratio of 8.1%. The filter had been fired at 900 °C and was found to remove more than 99.96% of *E. coli*.

When optimizing the filter composition, increasing the mass concentration and screen size of burnable materials (in this case sawdust), increased the permeability properties of the porous AgNP impregnated ceramic water filter pots and also decrease the bacteria removal efficiency. The high the amount and screen size of sawdust in the mixture the higher the porosity of the ceramic water filter, hence the lower the bacteria removal efficiency of the filters. In addition, the filtration and bacterial removal efficiency rate of the designed porous AgNP ceramic water filter in the study also slightly affected by firing temperature of the furnace. Increase in firing furnace temperature lead for agglomeration and sticking of particles of the clay soil material which decreases the porosity of the designed porous AgNP impregnated ceramic water filter pots. Therefore, this study finds that increase in firing furnace temperature will decrease water filtration rate and increase bacterial removal efficiency.

5.2. Recommendations

The following are recommended for further investigation:

- Comparison of other different burnout materials like rice husk and their effects on the microstructure of the filters and water treatment.
- Effect of compaction pressure on the percolation rate of water through ceramic filters.
- Effectiveness of the filters in removing heavy metals and other forms of ions from water.
- For how long AgNP will be effective and wear out from the porous ceramic water filter pots.
- The effect of thickness of the ceramic water filter pot should be investigated

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APPENDIX

Appendix A

Table 1.1: - Filtration and Bacterial removal test Result

		Factor 1	Factor 2	Factor 3	Response 1	Response 2
Std	Run	Percentage of Sawdust	Sawdust Particle Size	Furnace Temperature	Filtration rate	Log reduction of E. coli
		%	µm	(°C)	L/hr	CFU/100 ml
7	1	5	400 - 600	1100	1.15	4.434
17	2	10	400 - 600	1000	1.92	2.421
16	3	10	400 - 600	1000	1.95	2.348
10	4	10	600 – 800	900	2.43	1.237
2	5	15	200 – 400	1000	1.86	1.989
8	6	15	400 - 600	1100	2.32	1.275
5	7	5	400 - 600	900	1.35	4.882
11	8	10	200 – 400	1100	1.21	5.398
9	9	10	200 – 400	900	1.46	4.291
12	10	10	600 – 800	1100	2.29	1.021
3	11	5	600 – 800	1000	1.69	2.233
14	12	10	400 - 600	1000	1.93	2.534
4	13	15	600 – 800	1000	3.02	0.133
13	14	10	400 - 600	1000	1.96	2.501
1	15	5	200 – 400	1000	0.79	7.579
6	16	15	400 – 600	900	2.55	0.165
15	17	10	400 – 600	1000	1.95	2.466

Tebale 1.2: - Swamp surface Microbial removal efficiency test results

Run/code	Amount of e. coli on the source water (CFU/100ml)	Amount of e. coli on the filtered water (CFU/100ml)	Removal efficiency (%)
3	107	0	100
12	107	0	100
13	107	2	98.13
15	107	0	100

SEM PROCEDURE

✓ **Note the SEM is field emission electron microscope**

Model is inspect f50

1. Crush the samples into fine powder forms
2. Clean the sample holder specimens using acetone
3. Cut carbon graphite tips in small pieces and stick on specimens
4. Add the silver nanoparticle on specimens
5. Then open the scanning electron microscope by venting the vacuum.
6. Insert the samples into SEM using tweezers.
7. Pump the vacuum system to create under vacuum
8. Wait some minute till the vacuum pressure reaches 1×10^{-3}
9. Annotation the sample name
10. Click the beam on
11. Start to do surface morphology analysis by adjusting the breathiness, stigmatism, contrast, focusing, magnification, working distance, working voltage and spot size
12. Save the desired image what the researcher needs

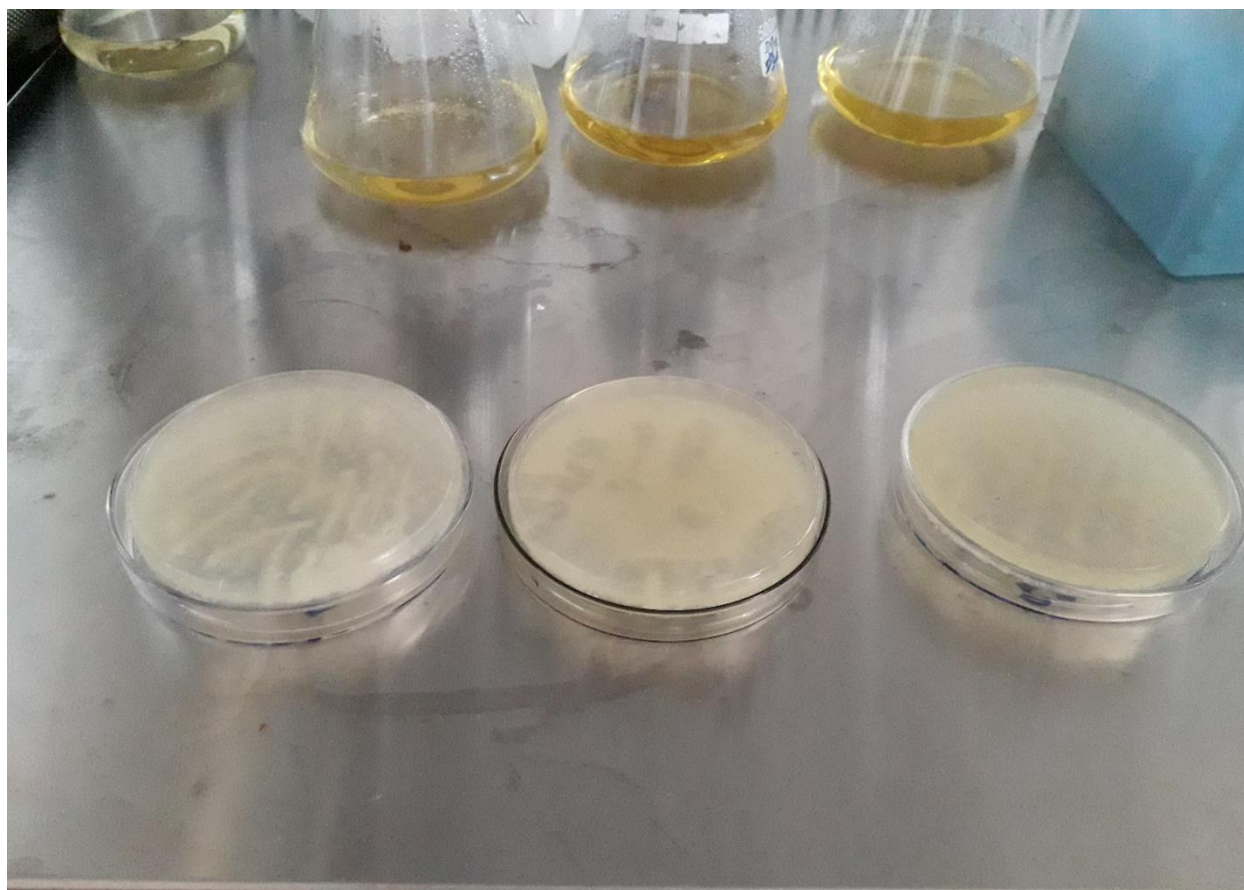
Appendix B: Lab equipment and samples



Furnace for Firing samples of CWFP



UV-Vis Spectra Photometer



Inoculated E. coli



Working on serial dilution at the laminar hood



Stirring sample



Pressing and mixing clay soil and sawdust to make ready for molding