FABRICATION AND CHARACTERIZATION OF CERAMIC MEMBRANES FOR SUGAR INDUSTRY

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CERTIFICATE

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DEDICATION

We would like to thank God for giving us this opportunity; thank our respective families and friends for the constant support; and our mentors, teachers, faculty for the guidance provided at every turn.

We would also like to thank ourselves for putting in the effort and teamwork.

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PREFACE

This work had been carried out with the aims of introducing optimized ceramic membranes for the use of the sugarcane refinery process. The proposed solution, subject to further optimization, was made to balance process efficiency and operation costs such that a cleaner, greener and economically feasible operation could be outlined, with the aims of improving the sugarcane industry in Pakistan. The core aims of this project were first to suggest Al_2O_3 ceramic membranes as the optimum filter for use in the sugarcane industry, after comparing different alternatives of polymeric as well as ceramic membranes; secondly, to optimize production of Al_2O_3 membranes to allow for industrial up-scaling of the process in the future; and lastly to reduce carbon footprints of replaced polymeric membranes through a cleaner and greener process.

ABSTRACT

A ceramic nano porous membrane of pure Al_2O_3 (with dimensions 2.54 cm diameter and 0.2 cm thickness) has been prepared via hydraulic pressing and subsequent sintering. Numerous samples prepared under different sintering conditions have undergone characterization via hardness testing using Micro Vickers hardness test, compression testing using Shimadzu Testing machine and pore size analysis through SEM; after balancing mechanical properties with porous open networks, an optimized sinter cycle of 1250°C with a soaking time of 15 hours has been suggested. Additionally, a lab-scale filtration apparatus has been fabricated for experimental testing of the sintered Al_2O_3 samples to simulate the process at lab scale while maintaining 5–8 bars of pressure. The filtration apparatus and filter can be optimized further for industrial usage and a simulated process flow has been proposed.

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INTRODUCTION

Although research into the viability and efficiency of using ultrafiltration for refining sugar started in the early 21st century, the process was patented in 1999 (14), which marked the advent of ultrafiltration being part of the sugar refining process in the food industry. In this paper, it was suggested that since the production of sugar from sugar cane is highly dependent on filtering the sucrose solution to refine it, the application of ultrafiltration during this step would be a better alternative to the process that was initially being used.

A study in May, 2000 dealt with the possibilities of applying microfiltration and/or ultrafiltration in the process of the purification of raw sugar juice. Since the processing of sugar has popularly been one of the most energy-intensive processes in the food industry, the application of ultrafiltration seemed to be all the more difficult. The only factors allowing ultrafiltration to be viable were the high volumes of juice being pumped at high viscosities, thus generating high pressures (15). This research also revised the findings of another paper in 1991, in which a unit of Alfa-Laval filtration with hollow-fiber modules embedded in it was used. This filtration unit, if subjected to recycling of raw sugar juice instead of a single-stage filtration stage, allowed for discoloration of raw sugar juice to up to 60–90% (16). Unfortunately, in the year 2000, despite the usage of ceramic membranes, the technology at the time impeded the usage of ultrafiltration at an industrial scale, as the findings of the same study suggest; this was primarily so because of the high price of ceramic materials along with the issues of fouling reducing the permeate flux and total performance of the filtration.

After the process of ultrafiltration was revised and inducted into the industry in 2002, the usage of polymeric membranes was commonplace. Polymers, being cheaper than ceramics to procure, as well as having much lower manufacturing costs, became highly

preferred in the sugar industry. But with the high versatility, lower cost and higher availability of polymeric membranes, there are several drawbacks as well. Thermal degradation, chemical degradation and increased fouling are to name a few of them. A study in 2004 was carried out using polysulphone membranes for ultrafiltration so as to study the behavior of flux decline of sugarcane juice with time. Different pore sizes were used at varying pressures (between 1.0–2.0 bar) and temperatures (50–70°C). Fouling was seen to be a major issue especially in the first interval of operation (17).

Additionally, with time, with technological advances being the pivot, the wheel of ceramic manufacturing turned to reach new heights, with ceramic manufacturing becoming easier and more versatile. The use of ceramic membranes for ultrafiltration at an industrial scale was soon realized.

Ceramic membranes have a higher ultimate tensile strength; are more inert and considerably more resistive to chemical attack; show lower rates of flux decline due to fouling and/or degradation; are more environmentally friendly, which can be backed by the study carried out in 2008 using a zirconium oxide membrane that was supported by an aluminum oxide layer used for ultrafiltration of beet juice (18); and have higher operating life than polymeric membranes.

This dissertation observes the use of ceramic membranes made of alumina for the use of sugar refining. It draws an argument as to how viable the replacement of polymeric membranes with ceramic membranes on an industrial scale could be, especially in the domain of the sugar industry.

LITERATURE REVIEW

2.1 History of Ceramic Membranes:

The journey of the invention of ultrafiltration began initially after the phenomenon of osmosis was discovered. The first publication about osmosis was by a French cleric by the name of J. Abbe Nollet: J.A. Nollet, Lecons de physique experimentale, Hippolyte-Louis Guerin and Louis-Francios Delatour, Paris, 1748. After this initial discovery, the following century especially focused on osmosis, particularly in the fields of biological and medical sciences.

After a lot of years following the discovery of osmosis, the German scientist, Dr. Adolf Eugen Fick introduced the first synthetic membrane that carried out osmosis at high pressures in 1855. Later on, the term "ultrafiltration" was coined by Dr. Bechold from Germany. Dr. Bechold defined ultrafiltration (UF) as the process of 'pushing multi-component fluids at extremely high pressures through especially prepared membranes of low porosity.' Consequently, the process of membrane desalination was introduced by Dr. Hassler in 1950.

During the thirty years following World War II, the French nuclear agency, the Commissariat à l'Energie Atomique, took on the responsibility to develop independent and civilian nuclear facilities so as to facilitate economic growth. The beginning of 1980's marks the first-time ceramic barriers were used for uranium enrichment by the process of gas diffusion. The leading group for academic research that was then being carried out into ceramic membranes was led by Professor Louis Cot at the National Graduate School of Chemistry in Montpellier. This particular group was responsible for creating a fully membrane-materials-dedicated laboratory by 1994, and also the creation of the European Membrane Institute of Montpellier by 2000. During the same time, several international companies accepted ceramic membranes for integration in filtration

devices, and therefore several companies were also dedicated to manufacturing ceramic membranes to meet the demand.

Since the high consumption of natural resources results in rapid depletion of the resources as well as the environmental damage inevitably caused by the burning of fossil fuels, ceramic membranes were brought into light for several processes. Ceramic membranes, due to their long working life and environmental friendliness, were preferred in technical, economic and environmental aspects. The membranes, being asymmetrical and multi-porous, come usually as multi-channeled structures that are composed of various materials ranging from zirconia to alpha alumina, including some mixed oxides that are interlinked in metallic housings to form composites.

The advantages of ceramic membranes include that the membranes are highly durable with high hardness. They can be used at high temperatures and even at extreme pH levels without degrading; high transmembrane pressures can also be used, making these membranes highly fit for ultrafiltration and associated techniques. This makes ceramic membranes essential for various applications in which polymeric membranes cannot be used.

The flexibility of ceramic membranes allows them to be available in several different pore sizes that are available in the commercial market, each membrane category designed and manufactured to meet the specific process needs. The membranes can be used for microfiltration, ultrafiltration and even nanofiltration, making them useful for applications ranging between 5mm to 1000 Daltons in size. This has resulted in the use of ceramic membranes being widespread in industries including the food, dairy, pharmaceutical, biotechnical, petrochemical, power generation, and beverage industries along several others. The dimensional attributes of the membranes used for each industry are tailored for their use in that industry.

The recent research on ceramic membranes is primarily focused on the development of ceramic membrane systems that incorporate either porous or dense membranes for use in fuel cells, reactor-separators and to extract components of extremely low concentrations from fluids at high volumes at near ambient conditions.

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2.2 Ceramic Membrane Types:

The primary categories of ceramic membranes are according to their structure – as dense or porous membranes:

2.2.1 Porous Membranes:

The defining characteristics of a porous membrane are its pore size, thickness and surface porosity. Out of these characteristics, the pore size of the membrane decides what application the porous membrane can be used in. These membranes are primarily used in solid-gas and solid-liquid separation.

Table 1 gives a summary of the different types of ceramic membranes differentiated by their pore sizes.

Type	Pore Size(nm)	Application
Macro-porous	>50	UF, MF
Meso-porous	2-50	UF, NF, GS
Micro-porous	<2	GS
Dense	-	GS, REACTION

UF: ultra-filtration
NF: nano-filtration
MF: micro-filtration
GS: gas separation

Table 1: Types of ceramic membranes

Porous membranes can be further classified into symmetrical and asymmetrical porous membranes; the membrane is called the former if the pores within the structure are nearly equally sized throughout, or it is called the latter when the pore size shows variation across the membrane structure.

The separation mechanism governing separation in porous membranes is that of molecular sieving.

Microfiltration membranes have found use in removing suspended matter and bacteria, the only limitation being that the removal of some microorganisms and of fully dissolved

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components is difficult. Therefore, nanofiltration appears to be a preferable option for this use, taking into account the smaller pore sizes.

2.2.2 Dense Membranes:

This category of membranes is governed by an extremely intricate permeation principle and technique for separation. Dense membranes are used primarily for gas separation. The process of separation that non-porous membranes follow is that the molecules that are easily permeable into the membrane first dissolve into it and then finally desorb out of the membrane.
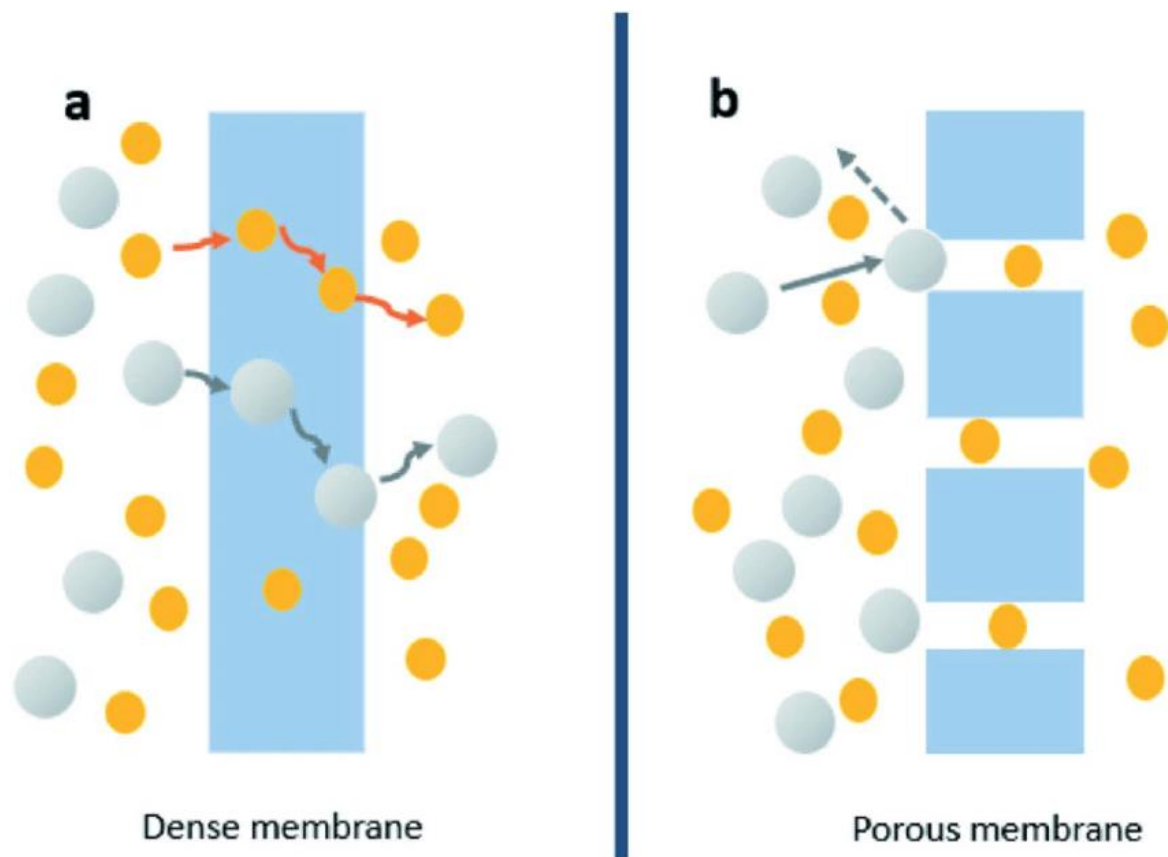
Figure 1: Geometrical configuration of membranes

The secondary classification of membranes is according to their geometrical configuration. These include the following:

2.2.3 Plate and Frame (Pillow-Shaped) Membranes:

Pillow-shaped membranes are made up of flat plates, the packing together of the two flat plates results in a pillow-shaped membrane unit, thus giving the membrane a name.

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Inside the pillow structure, there is a supporting plate. Several pillows can be placed at certain distances from each other, depending on the application; in the case of wastewater filtration, the water flows through the membrane and the permeate settles inside the space between the membranes.

Figure 2: Plate and frame ceramic

2.2.4. Tubular-Shaped Membranes:

This type of membrane is primarily used for fluids that are viscous or of bad quality. During filtration, the permeate passes through the membrane during solution flow and is collected in the tubular housing at its core, which also acts as a supporting layer for the membrane. These membranes are popularly used, considering their costs and efficiency. Generally, the diameters of tubular membranes range between 5 to 15mm. The figure below shows the geometrical structure of tubular / straw membranes.
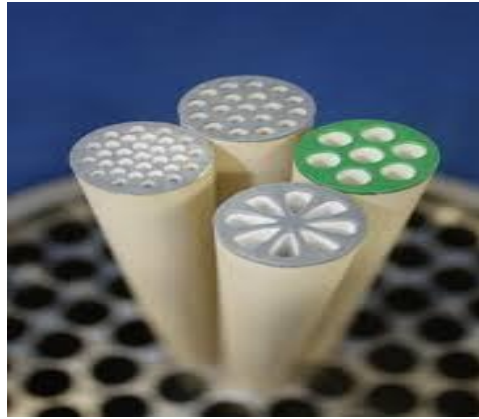
Figure 3: Tubular / straw membranes (diameter ≥ 5 mm)

2.2.5 Capillary Membranes:

Capillary membranes are built to withstand high filtration pressures and undergo filtration by acting as selective barriers. For this reason, the flow direction of capillary membranes can be either inside out or outside in. These membranes generally have diameters ranging between 0.5 and 5mm – much smaller than tubular membranes. But due to the smaller diameters, the risk of plugging increases considerably. The much higher packing density of capillary membranes serves as a compensation for plugging issues in this regard. The figure below shows the geometrical configuration of the membranes.
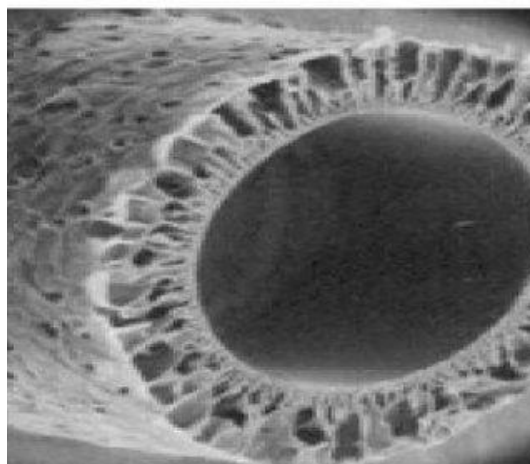
Figure 4: Capillary membranes (0.5 mm ≤ diameter < 5 mm)

2.2.6 Hollow Fiber Membranes:

These membranes have the lowest diameters as compared to the above geometric configurations – hollow membranes have diameters below 0.5mm. Therefore, the chances of plugging are relatively the highest out of the other configurations, and the packing density is subsequently the highest. Due to these properties, these membranes find specific use in the treatment of water containing low suspended solid contents.

Hollow membranes are used generally for nanofiltration and reverse osmosis (RO). During the process of filtration using hollow membranes, the permeate settles in a cartridge area surrounding the fibers as the feed solution flows through the spaces between the fibers. The figure below shows the geometrical configuration of hollow-fiber membranes.

Figure 5: Hollow Fiber Membrane (0.05> diameter)

2.3.0 Comparison with Polymeric Membranes:

Generally, the manufacturing costs of ceramic membranes is significantly high due to the expensive raw materials used in manufacturing. The raw materials include titania, zirconia and/or alumina. Due to this, the manufacturing cost of polymeric membranes is almost one-fifth the manufacturing cost of ceramic membranes; prices range between $100–200 per membrane to $1000–2000 per square meter in the case of special ceramic membranes which are used in harsh environments. The advantage of ceramic membranes here is that their operating life span is much longer than their polymeric counterparts; a ceramic membrane can last up to 23 years without replacement while a

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polymeric membrane's operating life is much lower. For example, a research found that the working life of polypropylene membranes in water filtration process was a maximum of only nine years.

Due to the latest advances in the manufacture of ceramic ultrafiltration membranes, the capital costs are similar to polymeric hollow fiber ultrafiltration membranes. Ceramic membranes show better mechanical properties, have an operating life that is much longer, and they overcome several operational limitations that arise in polymeric ultrafiltration membranes; ceramic membranes are able to withstand constantly high flux rates of cold water of different water qualities.

Traditional polymeric membranes used for microfiltration and ultrafiltration have found widespread use especially in industrialized nations. This is because microfiltration/ultrafiltration membrane systems have shown several advances that have resulted in decreasing prices, the most noteworthy of them being better manufacturing procedures and various technological advances that have resulted in higher packing densities and the hollow fiber geometrical configurations. The typical guarantee for the operating life of a polymeric microfiltration/ultrafiltration membrane ranges between 3–5 years for use in water treatment systems, and 7–10 years for systems that introduce harsher environments. In spite of several advances and prevention techniques used to increase the operating life of polymeric membranes, the issue persists.

Additionally, the constraints in the usage of polymeric membranes are that of operating temperatures: in 1988, Flemming F. Stengaard introduced a special coating to modify polymeric ultrafiltration membranes called the ETNA series, these membranes exhibited working temperatures approximately in the range of -3.89–25°C. Similarly, a research paper discussing the effect of high temperatures on polymeric membranes during the process of refining sugar juice, conducted in 2016 by Jianquan Luo, showed that the retention of the polymeric membranes including those made of polyethersulfone, polysulfone, polyamide (among others) started to decrease once the temperatures reached above 60°C. Polymeric membranes also show low resistance to harsh environments with solvents and other chemicals that degrade the polymer. For example, a study revealed that at lower pH values – pH 3 due to a hydrogenphtalat of potassium and HCl – a polymeric membrane deteriorated fast with the rate of fouling increasing

continuously over time. (2) They additionally exhibit low resistance to abrasion to sand or activated carbon. One of the major issues regarding polymeric membranes is that of fouling, which will be discussed later.

Due to novel production techniques as well as focused research into water treatment processes using ceramic membranes, there have been considerable advancements in ceramic microfiltration/ultrafiltration membranes, especially so in increasing surface areas and reducing prices. Goldsmith, 1988, invented a high-surface-area monolithic structure for ceramic membranes which resulted in a notable advancement that reduced the cost and increased the possibilities for the use of ceramic membranes in various applications. Another noteworthy development was seen by the company named Nanostone Water, Inc. which patented a new design (Göbbert and Volz, 2010) of a monolithic structure consisting of several individual ceramic layers that were potted together. This design highly reduced the production cost of ceramic membrane structure. Due to these advances (among others), the production costs of ceramic membranes are highly competitive with those of polymeric membranes.

Pretreatment is a necessity for polymeric membranes to show enhanced filtration properties that are usable for most processes. That said, ceramic membranes are operable with significantly less pretreatment in most cases; one example of pretreatment used for polymeric membranes is that of the elimination of the need of clarification systems – this reduces the initial capital cost, the cost during operation as well as the footprint. Additionally, ceramic membranes exhibit a higher total suspended solids (TSS) tolerance, which results in greater variability in the design parameters of any process using ceramic membranes. A high TSS tolerance will also allow for much higher system recovery rates if the process demands as such; the ceramic membrane would accordingly be tailored to the parameters set for the process at a flexibility much higher than that of polymeric ultrafiltration membrane systems.

Ceramic membranes also allow for harsher chemical and hydraulic cleaning methods without degrading or cracking, and there is a considerably lower risk of fouling as compared to polymeric membranes.

The coefficient of thermal expansion of ceramic materials becomes useful for ceramic membranes in the case of cold water flow systems below 5°C; in such cases, the pores

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within the ceramic membrane's structure would not show dimensional changes resulting from expansion or contraction, thus resulting in a stability for all operational parameters. There is no observable change in permeability except for the independent change in water viscosity. In the industrial domain, this means that plants can simply resize the feed pump to run at higher pressures during cold intervals so as to keep the plant output constant. Due to this, a competitive design flux can be maintained for as long as the process is ongoing, with the only parameter that would change would be the operating pressure, which would increase linearly with the viscosity of the water feed.

A research into the higher recovery rates of ceramic membranes was done when ceramic ultrafiltration membranes were comparatively evaluated with existing polymeric ultrafiltration membranes at an industrial wastewater reuse facility. What was seen was that polymeric membrane recovery rates on average were at 90%, so as to accommodate the changes in water quality caused by intervals where high suspended solids were loaded. When applying the same feed wastewater to a test system with a ceramic ultrafiltration membrane, a recovery rate of 97% was sustained. The table below reports the costs saved annually when ceramic ultrafiltration was used instead of polymeric ultrafiltration; the table takes into consideration the facility's cost of source water as well as the improvement in recovery rate of ceramic membranes compared to polymeric ones. Providing us with a complete overview and comparison of Polymeric Ultrafiltration Systems in relation to ceramic Ultrafiltration Systems

	Polymeric UF System	Ceramic UF System
Flow	2.3 million Gallons per Day	
UF Recovery%	90%	97%
RO Recovery %	75%	75%
Overall Recovery %	68%	73%
Source Water Cost	$1.58/1,000 Gal	
Annualized Water Costs	$431,130	$361,487
Annual Water Comsumption	273M Gal/ Yr.	229M Gal. /Yr.
Savings	x	44M Gal./ Yr. 569,643. / Yr.

Table 2 Comparison between Polymeric and Ceramic UF Systems

2.3.1 Fouling Issues:

Resulting from their properties, there are several downsides to using polymeric membranes for ultrafiltration. Among the general disadvantages of the higher energy costs of ultrafiltration, the main issues of ultrafiltration using polymers specifically are membrane cleaning and membrane replacement.

The need for membrane cleaning and/or replacement leads to the deterioration of the performance of the membrane, resulting in a flux that declines with time. The main reasons of flux decline are explained by concentration polarization and fouling of the polymeric membrane.

Research in 2002 shows that the phenomenon of fouling is governed mainly by biomass characteristics, surface chemistry of the membrane and solution and their interactions,

operating conditions and the characteristics (e.g. pore size, thickness, surface roughness etc.) and properties (e.g. wettability) of the membrane. The operational parameters affecting fouling are applied pressure, and the initial concentration and molar weight of the components in the feed flux. This is caused by the solute adsorping of the solute into the membrane's structure, changing the surface chemistry between the membrane and the solution.

There are two types of fouling; the first being fouling of the membrane surface, the second being fouling of the pores internally. The former type is reversible – chemical cleaners or back flushing solve this. Pore fouling, unfortunately, is fully irreversible. Therefore, research is mainly based on trying to prevent or at least minimizing pore fouling.

One particular subtype of fouling is through organic molecules, namely biofouling. Biofouling follows the following path: first, macromolecules entering into the membrane are adsorbed into it either on the surface or into the pores, leading to a layer formation on the membrane called the "conditioning film." Second, some of the flux permeates through without adhesion, the residue (in the case of biofouling is bacteria, including humid substances, lipopolysachirides etc.) then grows into different species, thus slowly excreting extracellular polymers and developing a protective film over themselves. Over time and ultimately, the membrane becomes irreversibly blocked once all the pores are, membrane cleaning being somewhat ineffective remedy. Another subtype of fouling is
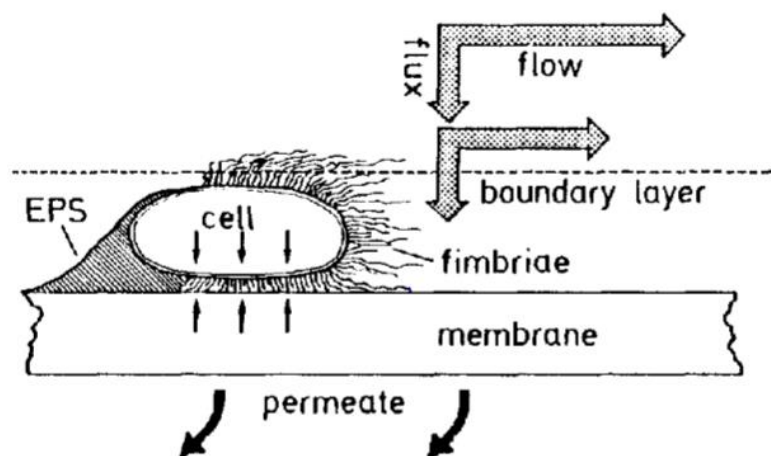

Figure 6: Schematic process of primary colonization (EPS: exopolymer substances)

through inorganic foulants. These foulants include magnesium, calcium or iron salts. A research regarding the characteristics of inorganic fouling compares the fouling rates of anionic polyacrylamide under different concentrations of carbon, sodium and calcium in the feed solution. This research concluded that divalent metal ions like that of Na^+ and Ca^{2+} played vital roles in membrane fouling.

Even though fouling is a major issue when considering polymeric membranes for ultrafiltration, there is extended research being done to make anti-fouling polymeric membranes or polymeric membranes with self-cleaning properties. One such research focused on inducing these properties on polymeric membranes by coating the membrane with titanium dioxide (TiO_2) particles using UV radiation on the membrane surface and interior structure. This coating on the membrane minimizes fouling of the membrane, thus increasing its performance.

Another research delved into four different membrane modification methods to reduce fouling. These included corona treatment, thermal treatment using PAAc graft polymerization and graft polymerization through HEMA, and modification with O_2-plasma. The final conclusion that this particular research venture drew was that the hydrophilic properties of PES membranes are considerably increased by plasma modification using O_2-plasma.

Surface coating techniques have been researched since a considerable amount of years now, one such research done in 1988 developed a new form of polymeric ultrafiltration membranes called the ETNA series. These showed much higher fluxes especially while processing protein-containing solutions. These also exhibited longer life spans of continuous operation and shorter cleaning cycles because of a lower rate of flux decline and better cleanability respectively.

Another example of research into improving the properties of polymeric membranes is one done in 1988 where polyacrylonitrile was grafted onto polyvinylchloride in a dimethylformamide solution using energy of gamma radiation from a Co6' source in the presence of an inert gas. The grafted polymer product obtained was separated. These exhibited better permeate flux flow rates and rejection. Another advantage of the grafted polymer produced was that it allowed for dry storage, an option previously impossible. That said, fouling remained an issue.
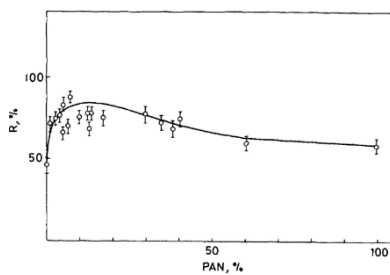
Figure 7: Rejection % 'R' vs. grafting % of PAN
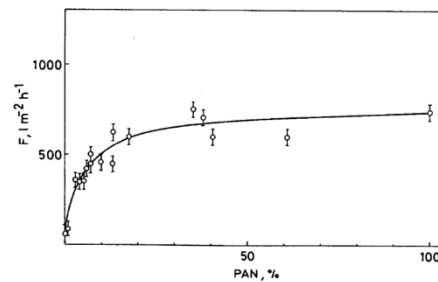
Figure 8: Permeate flux 'F' vs. grafting % of PAN

2.4.0 Material Selection:

A suitable filtration membrane for the use of ultrafiltration is one that is chemically and thermally inert, offers sufficient tensile strength and compressive strength, being one that is fairly easy to procure, while also being more affordable without compensating other properties. An additional consideration put into choosing the right material was that it would be easy to manufacture on large-scale, especially in a third-world nation.

Metals and alloys were ruled out of consideration due to their reactive properties, so the available options remained oxides, carbides and nitrides. Carbides and nitrides are considerably difficult to manufacture since they have high melting points and sintering temperatures, thus raising the manufacturing costs of these membranes. Oxides that were considered for use in this application were Alumina, Titania, Silica and Zirconia. Out of these candidates, zirconia was ruled out, because it was too expensive to procure and to manufacture. Titania and Silica were ruled out because they did not exhibit sufficient tensile strength and hardness levels for use in ultrafiltration processes. As a result,

alumina was the prime candidate that offered the best balance between, strength, toughness, hardness and high inertness. Thus, alumina was chosen as the material used in our research.

2.4.1 Alumina:

Identified as Aluminium (III) oxide, alumina occurs naturally in its crystalline polymorphic phase, α-Al_2O_3 as the mineral corundum. Due to its high free energy of formation, this material is highly chemically stable and refractory. Therefore, it is currently being used in processes with environments that are highly corrosive or have high temperatures. Further properties due to its high hardness lead to high wear and abrasion resistance. Subsequently, it is used in applications such as wear-resistant linings for pump and faucet seals, pipes and vessel thread.

Composition	Al_2O_3
Purity	Alumina 99.9%
Density	3.9gm/cc
Melting Point	2015℃
Specific Heat at 100 C	930J/kg K
Thermal Conductivity	40 W/mK at 20℃
Thermal shock Index	0.2
Flexural Strength	380MPa
Hardness HV	31500 Kg f/ mm²
Tensile strength	262 MPa
Poison ratio	0.26
Young's modulus	370 GPa
Co-efficient of Thermal Expansion	8 μm/ m ℃ Resistance Factors 1= Poor 5=Excellent
Flammability	3.9 gm/cc
Fresh Water	2015℃
Organic Solvent	930 J/kg K

Table 3: Properties of Alumina

Oxidation at 500C	40 W/mK at 20°C
Sea Water	0.2
Strong Acid	380 MPa
Strong Alkalis	31500 Kg f/ mm^2
UV	262 MPa

Table 3: Properties of Alumina (cont'd)

% AL_2O_3	Grain Size	Porosity	Applications Area
>99.6	Fine	Closed	Electrical
>99.8	Fine	Zero	Lamp tubes, Optical
>99.6*(recrystallized)	Medium	Closed	High temperature use
95-99.5	Fine	Closed	General electrical
80-95	Fine	Closed	Low duty electrical (spark plugs)
90-99.6	Fine/ Coarse	Open	Filter Media
80-90	Fine/ Coarse	Open	Abrasive

Table 4: Various Applications of Alumina

2.5.0

Methods for making Ceramic Membranes:

There are many techniques created for the fabrication of ceramic membranes, the major of these being: solid state casting, slip casting, dry pressing and extrusion. The steps generally followed for its preparation are:

1. Suspension preparation by proper mixing of the starting powder with a suitable binding liquid
2. The shaping of the prepared suspension according to a predetermined shape by a die.
3. Heat treatment to remove the binder and properly bond the particles by sintering and cause grain growth.

2.5.1 Solid State Method:

The solid-state method is one of the oldest and simplest methods for ceramic membrane synthesis. In this, the initial powder material – oxides, carbonates, or salts – are mechanically mixed, followed by heat treatment at high temperatures at above 1000 °C for a specific time depending on the density and grain size required. The extended heat treatment allows the diffusion of cations and anions in the solid state across the grain boundaries, ending up with the formation of the final ceramic product.

According to the concept of solid-state reaction, it is always estimated to have dense ceramic membranes. And, by using pore generators, porous ceramic membranes can be prepared by solid state processing of ceramic powders.

An example of a schematic processing route for the solid-state method to manufacture ceramic membranes is given below:
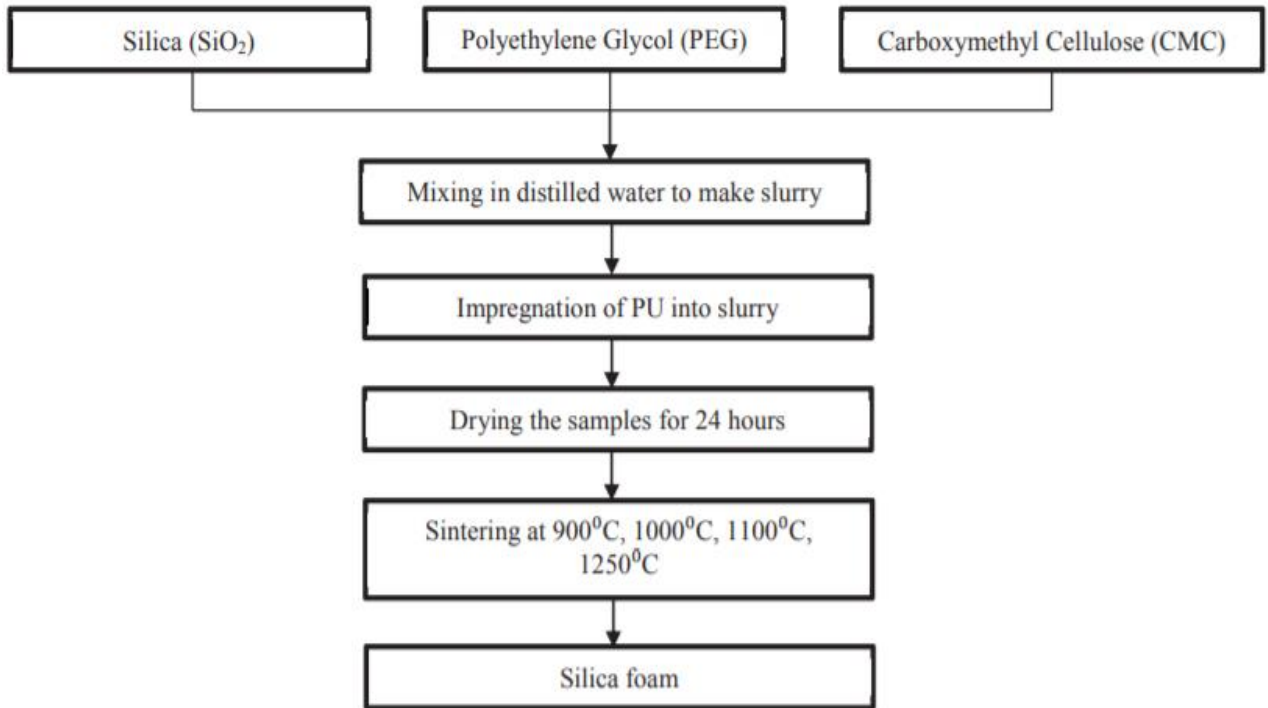
Figure 9: Solid State Method Process Flow Chart

2.5.2 Pressing Method:

In this method, a fixed amount of powder material and binder is poured into a die. After being punched or semi-punched at a given temperature, the mixture is sintered at a certain temperature for the necessary grain structure and pore size to be obtained. The prepared circular, plate-shaped and porous ceramic membrane is supported by dry pressing. An applied force is used to produce a sintered dense layer using a press machine which applies pressure higher than 100 MPa to produce a disk having thickness of about 0.5 mm to 2 mm. The support is with porosity up to 90%, pore size between 100 and 50 nm, and the degree of acid/alkali resistance over 98%.

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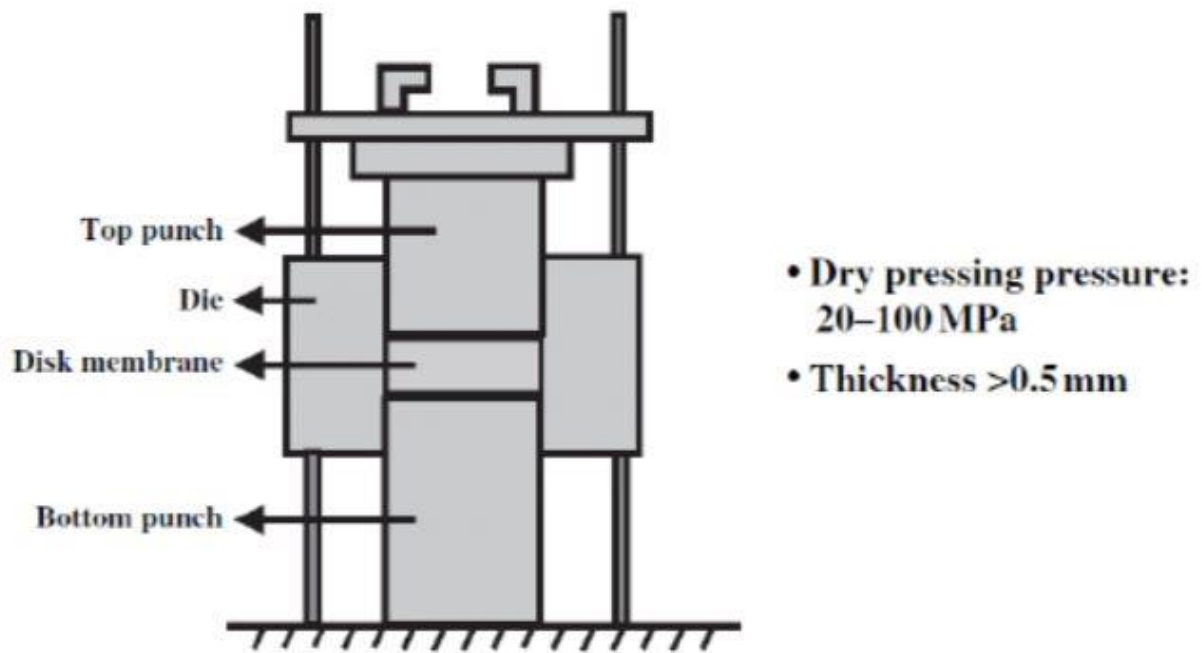
Figure 10: Dry Pressing Machine

2.5.3 Extrusion Method:

The extrusion method is a simple, mass-producible method for the manufacture of ceramic membranes that can be used for microfiltration/ultrafiltration. It finds its applicability mainly in the manufacture of porous ceramic tubes. The process of manufacture is by forcing a homogeneous stiff paste through a die (also called nozzle) to compact the material and shape it to form the final membrane. The membrane produced has sufficient green strength to withstand operation. Any remaining binders, plasticizers and solvents are evaporated from the green membrane so as to ensure that the membrane stays in the desired shape – the shape it has been produced as. The die determines the porosity, shape and pore size distribution of the produced membrane. The figure below shows the process of manufacturing ceramic membranes by extrusion method:

- Diameter: >2 mm
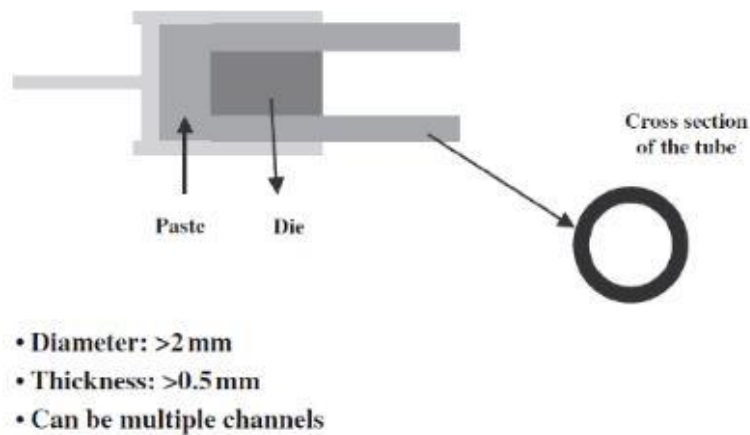- Thickness: >0.5 mm
- Can be multiple channels

Figure 11: Extrusion Method

2.6.0 White Sugar Refining Process:

The production of sugar consists of two major steps:

1. Obtaining raw sugar from the processing of sugarcane or sugar beets.
2. The process of refining sugar from raw sugar.

The extracts from sugarcane or sugar beets include sucrose, which is the concentrate that is to be extracted out. The impurities include lignins, proteins, polysaccharides, starches, waxes, gums, and other colloidal substances. These unwanted substances change the color and/or taste of the product, they also cause the product yield to be reduced.

Sucrose is the primary constituent of sugar. It can be hydrolyzed in an acidic solution, producing glucose and fructose. This can be observed in the equation below:

Sucrose + H2O → Glucose + fructose
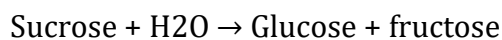
The steps for sugar refining process are as follows:

2.6.1 Step 1 – Affination:

Affination is the process in which the raw sugar obtained from the first major step is mixed with a saturated syrup which softens the film of molasses that have adhered to the raw sugar. The solution thus resulting undergoes centrifugation, so as to remove as much of the film of molasses as possible and filter out crystals that are more refined. During this step, the molasses dissolve into the syrup and are removed. The removed "impure"

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sugar syrup still contains some amounts of concentrate, so it is recycled separately in a boil-out section. The sugar thus recovered by boil-out are then sent back for re-melting them with the washed sugar. The impurities that are removed are later concentrated to produce molasses. Molasses are the by-products of the process of sugar refinery and can be used in brickmaking, for casting sands and others. The centrifuged sugar is utilized, maintaining a certain temperature and density of the molten sugar fluid. Finally, the sugar solution is screened to filter out any fibrous materials left in it.

2.6.2 Step 2 – Carbonation:

During the process of carbonation, the sugar from the solution is re-dissolved and an addition of calcium hydroxide (milk of lime) and boiler fuel gas (carbon dioxide) is bubbled into the mixture to produce lime and water as per the following reaction under controlled conditions:

$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$
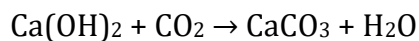
Unnecessary gum and amino acid impurities undergo precipitation and are removed with the calcium carbonate from the sugar syrup. Excessive color is also removed in this step.

The step of carbonation process is optimally carried out in two major steps so that the produced precipitate is of sufficient quality for filtration – the precipitate produced is of suitable size and has sufficient distribution of the precipitate particles. The residual lime content within the produced solution can be indicated by measuring the electrical resistance of the resulting solution. In the first stage, 80–90% of precipitation is recommended. The second stage revolves around the pH value of the solution, which is measured and controlled since it primarily governs how successfully the lime precipitates. If the pH value is below 7, the sucrose is hydrolyzed, and glucose and fructose are produced. Alternatively, if pH value is more 9, the sucrose undergoes alkaline destruction and results in colored components within the solution.

2.6.3 Step 3 - Char filtration:

During this step, activated charcoal (activated carbon) is added to the sugar solution, further removing color and inorganic impurities.

The calcium carbonate precipitate accumulates most of the impurities and is removed using filtration at high pressures. The membrane is a polypropylene filter cloth; the calcium carbonate as a filter aid. The sucrose residue is removed by water washing of the filtered mud. The filter mud is now considered as the waste material. More sweet sugar is then recovered by washing the waste material (mud) and is used for melting in boilers for earlier stages.

After the filtration, the solution produced is of a color similar to pure honey. This is called "raw liquor", which then undergoes another stage of decolorization by keeping it in contact with bone charcoal. Bone charcoal is activated carbon settled over a skeleton of calcium phosphate. The surface area of bone charcoal is high, which affords it a higher area of contact for it to absorb both color and inorganic ash impurities from the sugar solution. Following this decolorization step, the bone charcoal is rejuvenated using two steps: first, it is washed using water to remove inorganic impurities, and then it is heated in vacuum at 650°C so that volatile organic impurities are removed. The final, decolorized "fine liquor" is, at this stage, ready for the final step – to refine it fully and recover any further concentrate from it.

2.6.4 Step 4 – Crystallization:

In this step of crystallization, the sugar solution is boiled in vacuum to produce crystalized sugar crystals that are in a more usable form while removing any unnecessary impurities from the solution. Vacuum conditions are achieved using air pumps and vapor contact condensers.

This step is carried out at a lower pressure between 75–90 KPa and a lower boiling temperature between 60–70 °C. These parameters ensure that colored compounds are not formed. The "fine liquor" is concentrated until super-saturation is reached. Then, a small quantity of sucrose is added as seed to initiate shock-seeding and give spontaneous nucleation of sucrose precipitates of a fine nature. During the boiling, the growth of the crystal precipitates is fully controlled so that the particulate sizes of the crystal produced are optimal. The produced sugar is then graded before packing it. Any syrup being recycled goes through three further steps of recovery boiling. Contained in the final sugar produced, there are several components in the sugar solution:

- Golden syrup,

- Treacle,

- Raw sugar,

- Soft brown sugar,

- Coffee crystals,

- Castor sugar,

- Liquid sugar,

- 1A sugar,

- Varying amounts of glucose,

- Varying amounts of fructose,

- Low concentrations of inorganic impurities.

Some of these constituents can be further processed to produce other sugars, namely coffee crystals, golden syrup, soft brown sugar and treacle.
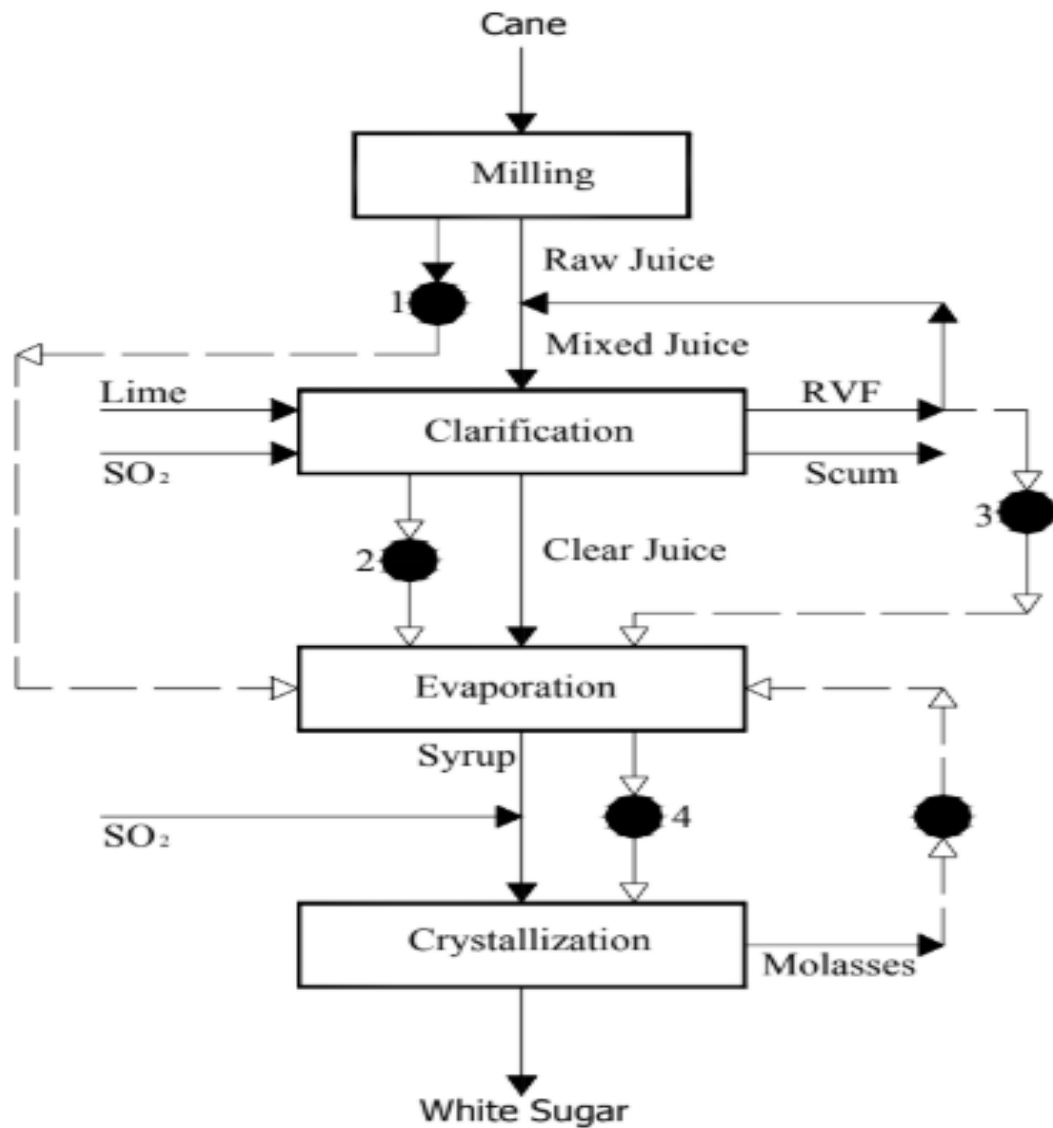
Figure 12:Conventional Sugar cane refining process flow chart

2.7.0 Use of Ceramic Membranes in Sugar Refining Process

The process of refining sugar is highly energy intensive. Therefore, the application of membrane technology for ultrafiltration presents a considerably viable option. The few limitations of this option include that since there is a high osmotic pressure and the high viscosities of the sugar solution, the membranes for filtration can only be used for dilute streams during clarification and purification, which occurs particularly at the stage of juice extraction.

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In the diagram below, the dotted areas in the process are those where ultrafiltration or microfiltration could replace the operation. During these stages, ultrafiltration and microfiltration can allow for efficient removal of colloidal and macromolecular impurities without any requirement of adding lime, carbon dioxide or sulphite to aid the evaporation and crystallization of the sugar solution. Even more so, not using macromolecules and reducing lime levels limits the issues of fouling and scaling in the evaporators.
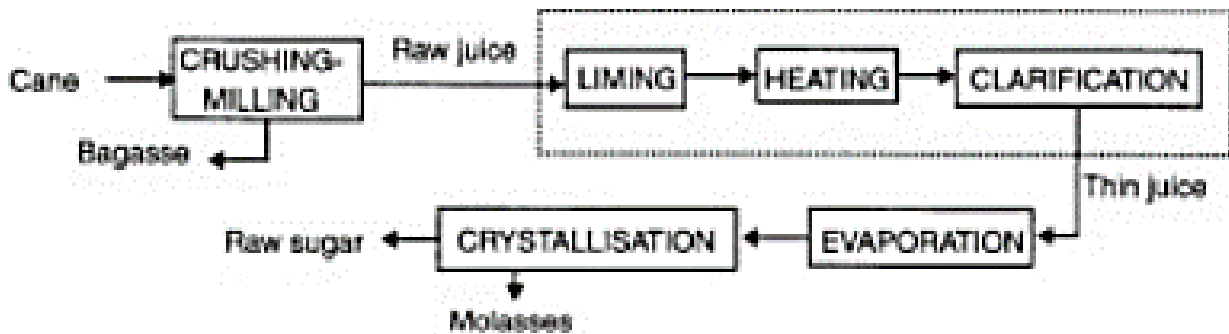
Figure 13: Sugarcane juice refining process using ceramic membranes

EXPERIMENTAL PROCEDURE

Rig Fabrication

AutoCad Design

Teflon Procurement

Lathe Machining

Finished Rig

Pallet Fabrication

Alumina Powder

Palletizing

Sintering

Characterization

Not Achieved

Achieved

Optimized Pallet

SEM

Compression testing

Vickers Micro Hardness

Final Testing

Rig and Pallet Setup

Testing

Brix

Flow Rate

Lifespan

Figure 14: Execution Plan

3.1.0 Die Design:

The first step of the whole manufacturing process of the ceramic filters is to manufacture a die from which pellets will be produced. Before material selection and fabrication of the die can begin, a 3D CAD design will be made, and the dimensions are finalized.
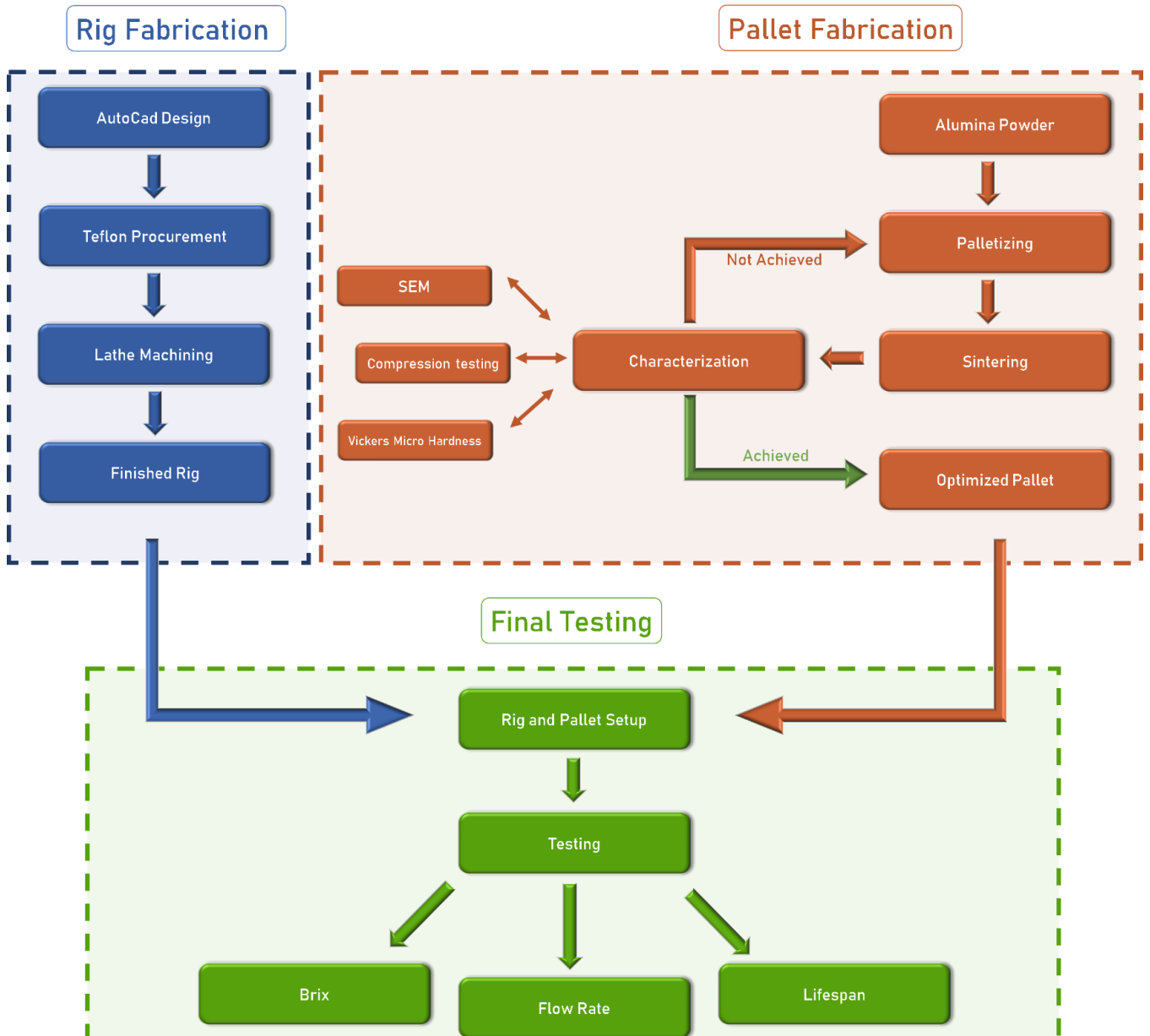
3.2.0 Material Selection and Die Fabrication:

Once the die, plunger and stopper are designed according to the specified dimensions, the material of the die fabrication is selected, which in this case is mild steel.

Generally, for dies tool steel is used but tool steel is expensive. Therefore, to stay economical while keeping the material limitation in mind i.e. the elastic modulus; mild steel is the material of choice. A chunk of mild steel is then machined using a lathe machine to create the three components i.e. die, plunger and stopper with minimal tolerances for a tight fit.

3.3.0 Pelletizing:

It is the first step for the fabrication of the ceramic filters. The die is positioned on the hydraulic press and silicon paper is attached to the face of the die stopper and plunger A weighed mass of 1.5 grams of 50 nm Alumina is loaded into the die cavity and slightly shaken to settle the powder in the cavity the plunger is fixed on top, and a uniaxial hydraulic force is applied for a limited time and the load is removed, then the die is turned upside down and the plunger will be pushed through to bring out and remove the pellet.

Figure 15: Sintered Pellet

The deciding factors for the green strength of the pellet are:

i.   The Hydraulic Pressure Applied:

Higher pressure will result in greater densification of the final compact with a smaller pore size; however, leading to a curvature being created, due to greater force acting in the center compared to the outer diameter of the pellet.

ii.   Loading Time:

The longer the time the load is being applied on the powder, higher and more uniform will be the densification of the final green compact formed.

3.4.0 Sintering:

Once the green compact is formed, the next step is to partially sinter the compact to achieve the desired densification/pore size.

Pore size is dependent on two factors:

Figure 16: Muffle Furnace

i. Sintering Time:

Sintering Time is directly proportional to densification and to achieve the desired pore size of 65 nm-85 nm at the surface and 120 nm-140 nm at the center of the pellet an optimized sintering time of 15 hours should be used. If excess time is given the sample may become brittle and break on minimal impact if the sample is not sintered for long enough it may lack structural integrity.

ii. Sintering Temperature:

Sintering Temperature is also directly proportional to densification but also acts as the activator for necking and the sintering process. To achieve the above-mentioned pore size an optimized temperature of 1250°C should be used. If the temperature is lower than specified; necking is insufficient leading to larger pore size and lower structural integrity and if it is above the specified range; agglomerates start to form leading to small pore size and induction of brittleness in the sample.

3.5.0 Characterization:

3.5.1 SEM:

To know the pore size of the sample, Scanning Electron Microscopy has to be performed. Before performing SEM, the sample has to be prepared accordingly. Metallic vapor is physically deposited onto the sample through a process known as sputtering to make the ceramic sample conductive. The sputtered sample is then placed inside the SEM which produces a 3-D image of up to 40,000x magnification. The pore sizes at the face and cross section are then measured using a software. In this case if the pore size is within the aforementioned ranges for the surface and cross section; it indicates that the pelletizing and sintering cycle has been optimized.

3.5.2 Compression Testing:

Each of the sintered sample is then tested for compressional strength to stimulate and study the effect of applied pressure during ultrafiltration. To test the sample, it is placed in a Universal Testing Machine which is programmed to compress the sintered product at a rate of 0.05 mm/min or at 2.5% strain per minute, as the sinter is about 2 mm in size. The resultant maximum stress from this test is the amount of pressure the pellet will be able to sustain before failure during ultrafiltration.

Figure 17: Universal Testing Machine

3.5.3 Vickers Micro-hardness Testing:

To determine the hardness of the sample, the procedure is to carry out Vickers Micro Hardness test as any other hardness test such as Brinell or Vickers would lead to failure due to the brittle nature of the sample.

Before the sample is tested for hardness it must be grinded to make sure the base of the sample is flat. While the face of the sample to be tested should be polished using a diamond suspension of 1 or 3 micron in size. To carry out the test, the sinter is placed on the base of the equipment, a microscopic lens of 20x is used to focus the surface of the sample. The focus on the sample is then fine-tuned using a 40x microscopic lens. Once properly focused a force of 1 kg is applied with a dwell time of 10 seconds using the diamond shaped indenter. The diagonals are then measured using the same 40x microscopic lens in both x and y-axis. Once the diagonals are set the apparatus calculates and prints the value of hardness of the sample according to the H_{V1} scale.

Figure 18: Vickers Micro-Hardness Tester

3.6.0 Fabrication:

3.6.1 Sinter Holding Device Design

Similar to the designing of the die for pelletisation, a device is designed to have the dimensions to ensure the sinter is properly fixed at its position, creating a near perfect seal. To achieve the optimum flow rate in both directions, this has to be ensured without dislodging the sinter from its position. The device consists of two parts, the top, and bottom, which need to be designed separately as single parts. The software used for the design is Autodesk Autocad version 2018.
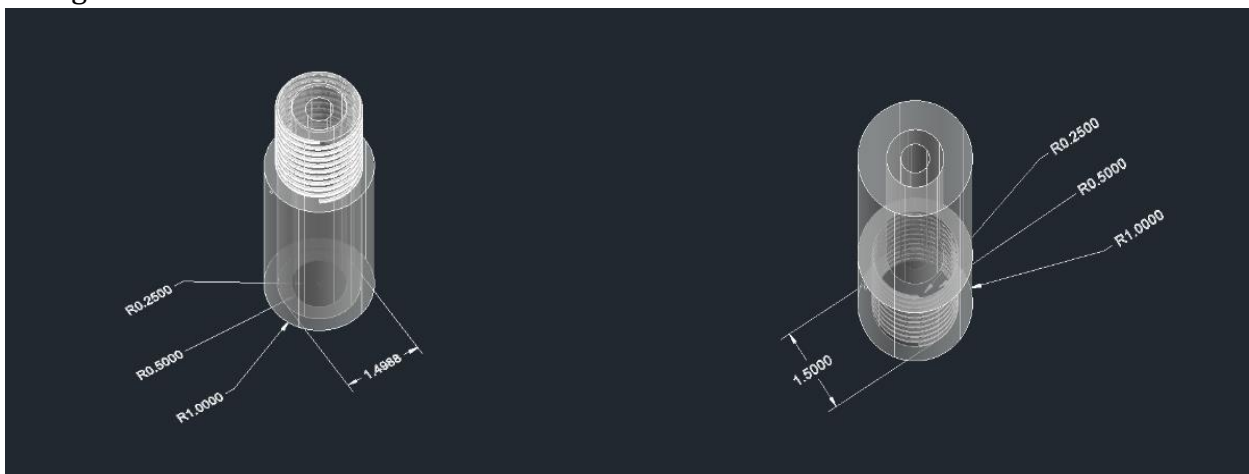

Figure 19: AutoCad Conceptual Drawing (top and Bottom)

For ergonomic purposes, grooves are added to the body of the device so that the process of placing/replacing a filter by screwing/unscrewing the device is easier and a proper grip can be maintained.
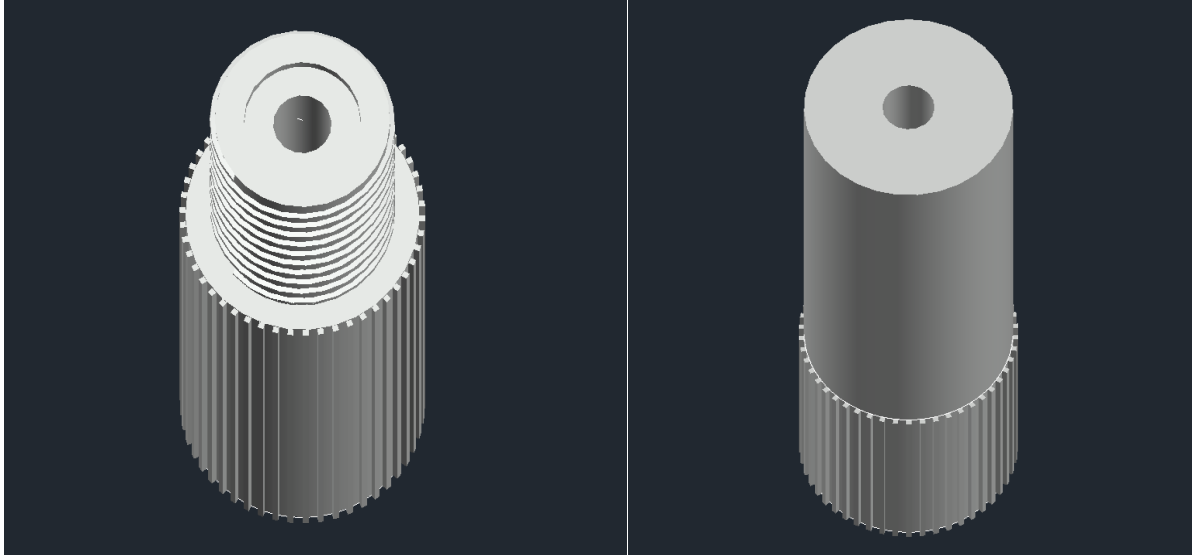

Figure 20:Finalized Pellet Holding Device Design

3.6.2 Pellet Holding Device Fabrication

After completion of CAD designs, a suitable material is selected for the fabrication, in this case, Teflon. It offers the necessary rigidity and strength while still being affordable and easily machinable.

The two parts of the device are fabricated separately on a lathe machine, giving minimum tolerance finish so no leakage can occur during testing.

Figure 21: Teflon Fabricated Pellet Holding Device

For future purposes, and once the fabrication of the sinter holding device can be industrialized using a more automated process, grooves are to be added to the body as discussed in the rig design (section 3.6.0). Considering that filters will have to be replaced after a certain period, albeit a lengthy period, of time, the grooves aim to make the process of unscrewing to remove the expired filter, inserting a new pellet, and then screwing back the rig and tightening it to ensure a perfect seal. Airtightness, as mentioned before, is a primary concern for efficient working of the device in the process of ultrafiltration.
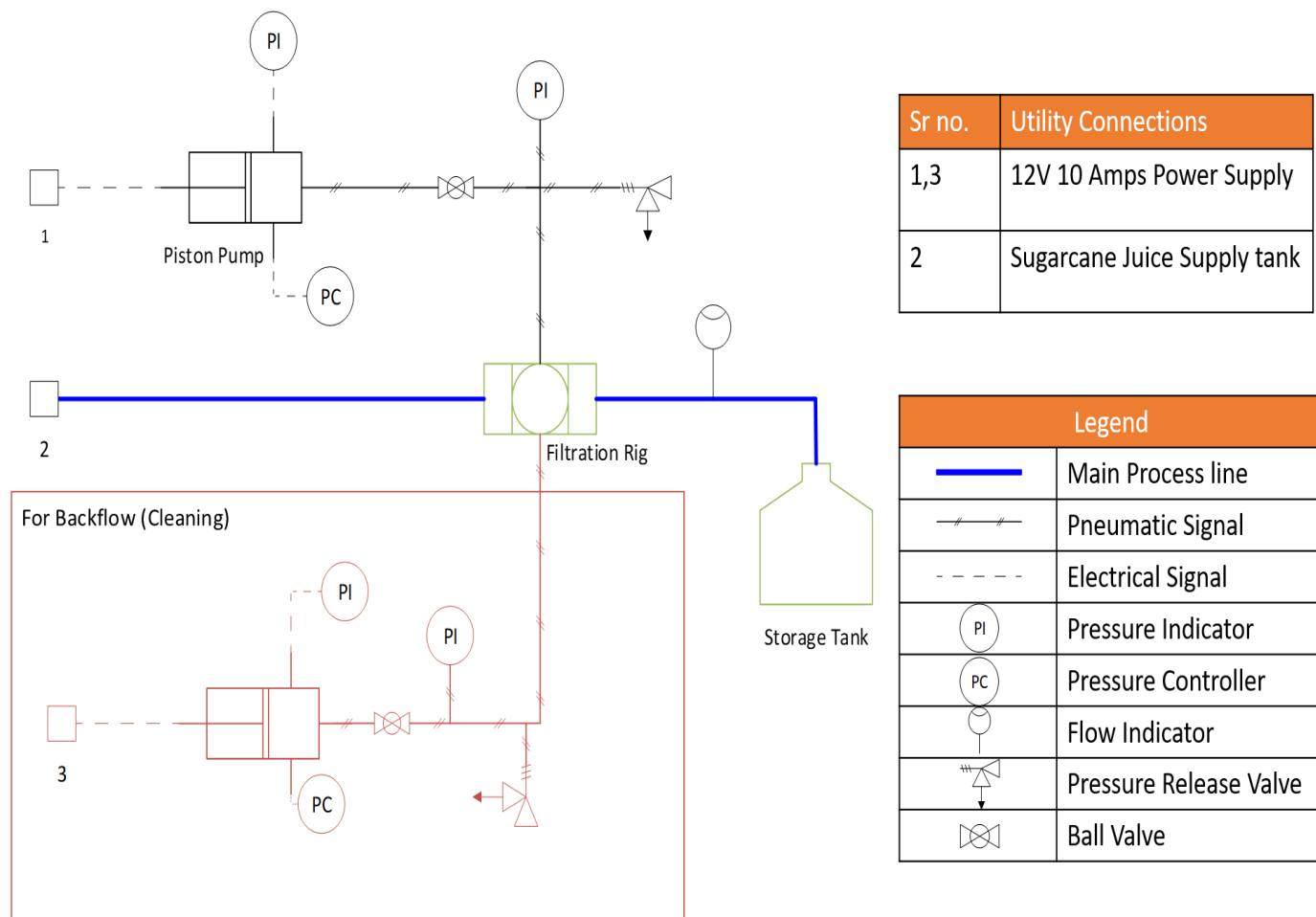
3.7.0 Setup

Sr no.	Utility Connections
1,3	12V 10 Amps Power Supply
2	Sugarcane Juice Supply tank

Legend	
————	Main Process line
—//—//—	Pneumatic Signal
- - - - -	Electrical Signal
PI	Pressure Indicator
PC	Pressure Controller
	Flow Indicator
	Pressure Release Valve
	Ball Valve

Figure 22: P&ID Diagram for the Rig Setup

3.7.1 Alumina Filter:

The 25.4 mm diameter, 2 mm thick Alumina pellet is manufactured by pelletizing followed by sintering (discussed further in section 3.3.0 and 3.4.0) into a final product. After being processed, the pellet undergoes standard tests such as SEM, compression

testing and Vickers Micro-hardness testing (discussed in section 3.5.0) to guarantee mechanical soundness. The pellet is the core of the entire setup; it is used as the filter in this ultrafiltration process. Since pressures can range between 4–8 bars, mechanical soundness is a fundamental requirement for reliable working. Another requirement is anti-clogging and efficient filtering, which the sintered, porous and inert ceramic filter affords us. The filter is indicated in the process flow diagram.

3.7.2 Filter-Holding Device:

Holding the filter is a filter-holding device, the details of which have been discussed in section 3.6.0 and 3.7.0. The purpose of this device is safe accommodation of the pellet, while ensuring air tightness and leak-proof operation at high pressures. The device is shown as a yellow cylinder in the process flow diagram.

3.7.3 Electric-powered 12V 10A Air Compressor:

To be able to generate the necessary amounts of pressure (4-8 bars), an air compressor pump of sufficient power is needed. The electrical air pump, labelled in the diagram, used in the setup is powered through a 12V, 10 A in the current setup at a laboratory scale. As a result, a low amount of power is sufficient and the lab-scale apparatus is portable.

The air compressor is digitally controlled with a digital pressure included; if any pressure loss is detected in the pipeline, the compressor corrects it by itself to maintain steady pressure throughout. Although a digital pressure indicator is included in the features of the air compressor used, the setup has incorporated another pressure gauge, the functionality of which will be discussed later.

3.7.4 Forward-Flow Operation

During forward-flow operation, the unfiltered solution is pumped through the inlet pipe, with the other end of the pipe connected to a container (for the apparatus at lab scale, the container is a measuring cylinder). Pressure is then applied using the air compressor with forward valve (ball valve 1) open and the backward valve (ball valve 2) closed, the resultant direction being forward (pressure being applied with the unfiltered solution on top of the alumina filter) and towards the storage container.

A pressure gauge has been added into the instrumentation of the apparatus, the purpose of it being to monitor pressure in the system, thus helping to maintain the pressure at a controlled value within the piping lines of the system. In the case of overpressure conditions, a rotating valve is in place as a pressure relief mechanism. This serves for the safety of the users, the equipment and the prevention of any losses pertaining to leaks and operation failure.

3.7.5 Backward flow Operation

Over time, the debris of residue would start filling the pores of the filter. If left unchecked, this will result in undesired drops in flow rate. To prevent this, a backflow mechanism may be suggested; the air compressor is connected to a pipe leading to the forward valve, but it splits before the valve towards a backflow loop, which is to be incorporated into the setup for routinely cleaning of the filter. Referring to the process flow diagram above, the backflow loop is highlighted in red. It is a beneficial feature especially to increase working life of the filter and to prevent clogging. To perform a backflow cycle, the air compressor – along with the rest of the operation – is shut down; the forward valve, which is the valve normally open during forward-flow operation, is closed. To prevent contamination of the process, the joint between the main piping and the storage container, as well as the solution inlet must also be closed. Instead of the forward valve, the backward valve is opened. This results in pressure being applied through the pipeline connected to the forward valve, across the tee joint fitting and onto the process input (unrefined sugar cane juice or impure water); it is applied in the opposite direction of the rig.

During backflow operation, pressure is applied through the backflow valve and the piping highlighted in red through to the piping tee joint below the rig. The solution flows up the rig and is removed through exit valve.

3.7.6 Sealants Used

Silicon is being used for airtight fixation of filter, to ensure that the unfiltered solution flows directly through the alumina filter and not through the crevices between the alumina filter and the holder at the point of fixation.

Hose clamps are used for the installation of rubber pipes in the system.

Teflon tape has been used at any point where there is a joint in the steel fittings.

RESULTS AND DISCUSSION

4.1.0 Material Testing

4.1.1 SEM

SEM was used to carry out pore size analysis for all samples created under different conditions. The face and cross-section scans of multiple sinters made under the same sintering conditions were analyzed and the average pore size resulting from each sintering conditions was measured. SEM analysis also helped observe any signs of interconnectivity between pores that resulted in an open network of porosity. To obtain higher resolution of SEM results on the relatively non-conductive nature of the ceramic product, sputtering times were kept up to half an hour for each sample. The left face and cross-sectional results of each scanned sample are shown below:
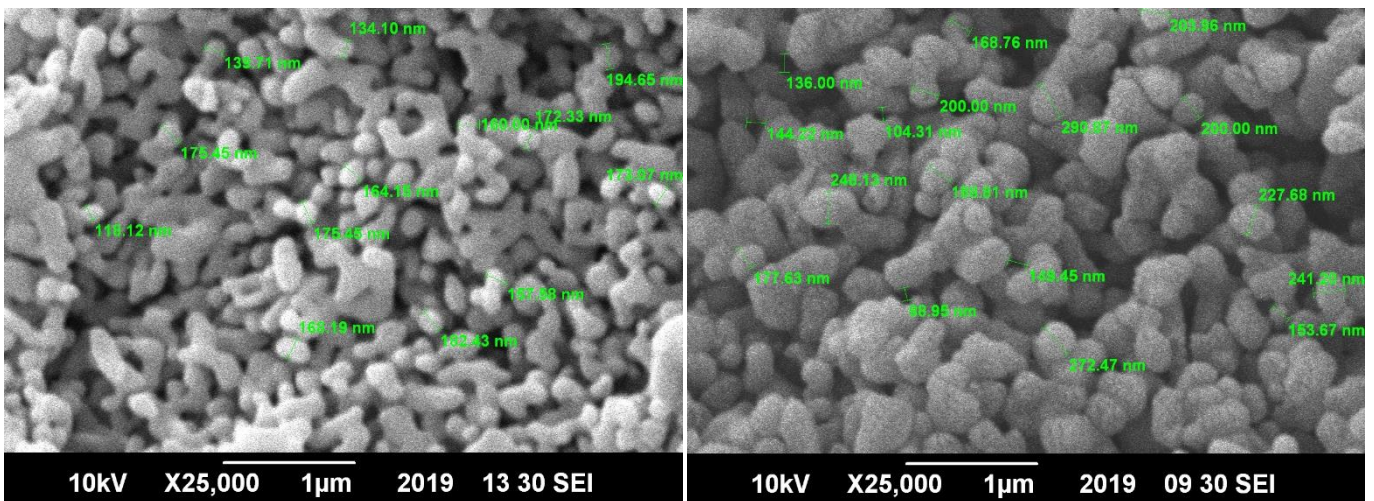
Figure 23: SEM of 1200°C 5 hours (left: face Right: Cross section)
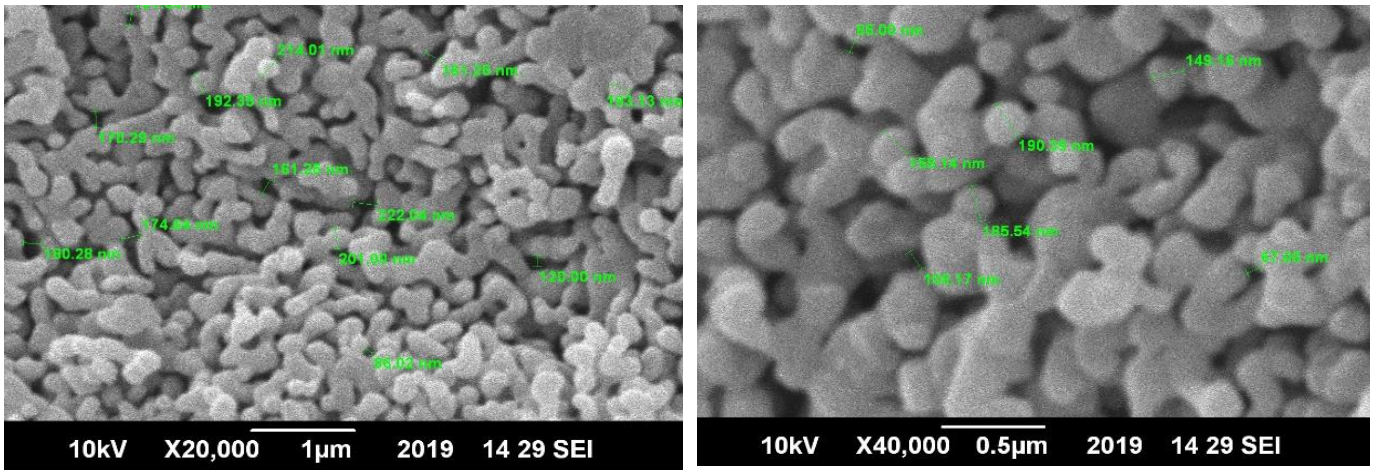
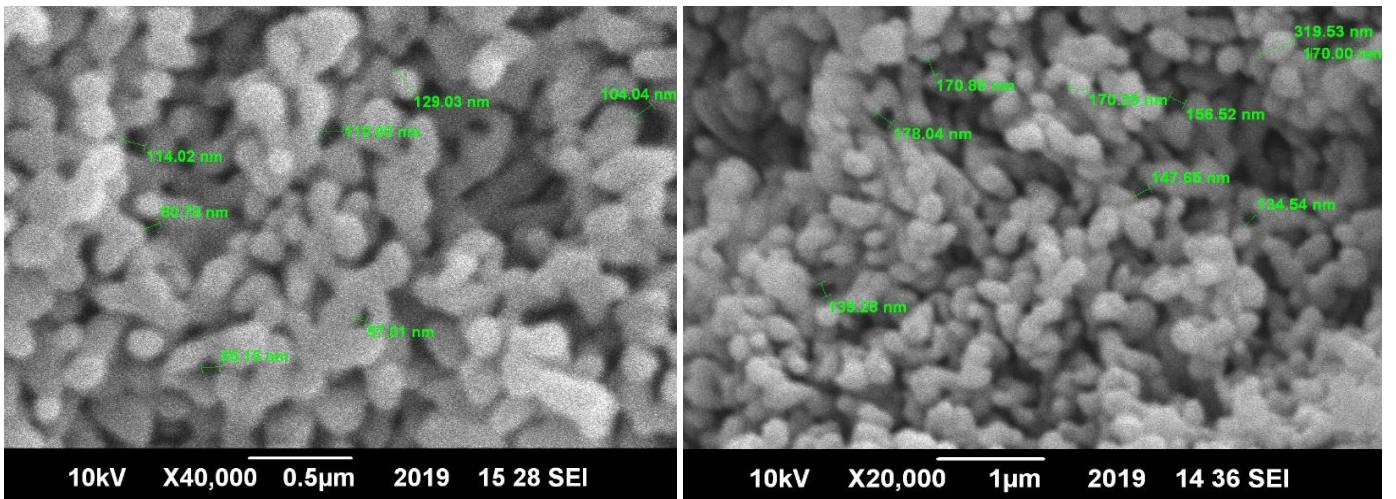Figure 24: SEM of 1250°C 5 hours Sample (left: face Right: Cross section)

Figure 25: SEM of 1250°C 10 hours Sample (left: face Right: Cross section)
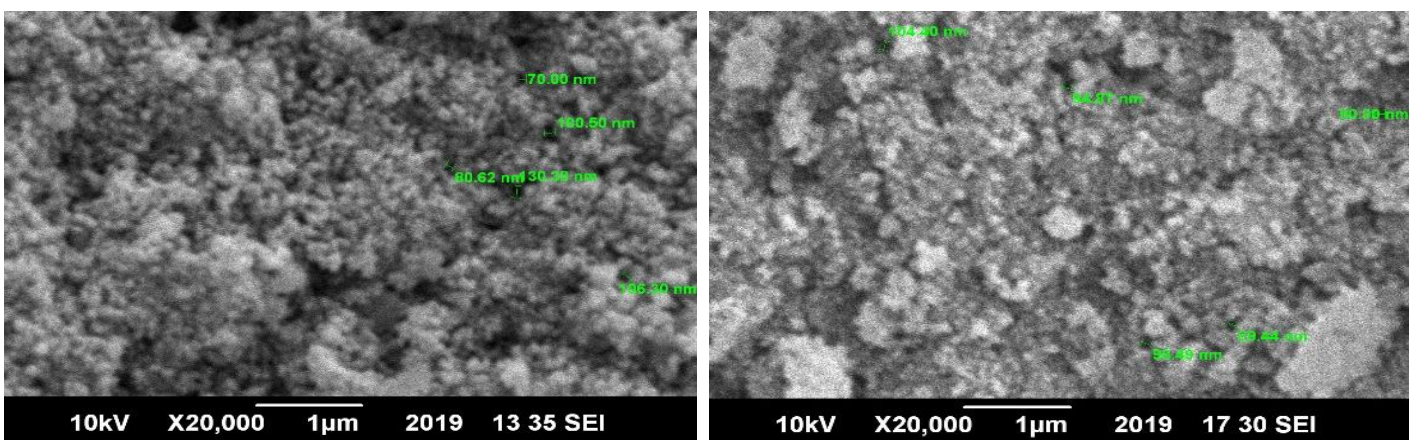

Figure 26: SEM of 1300°C 5 hours Sample (left: face Right: Cross section)
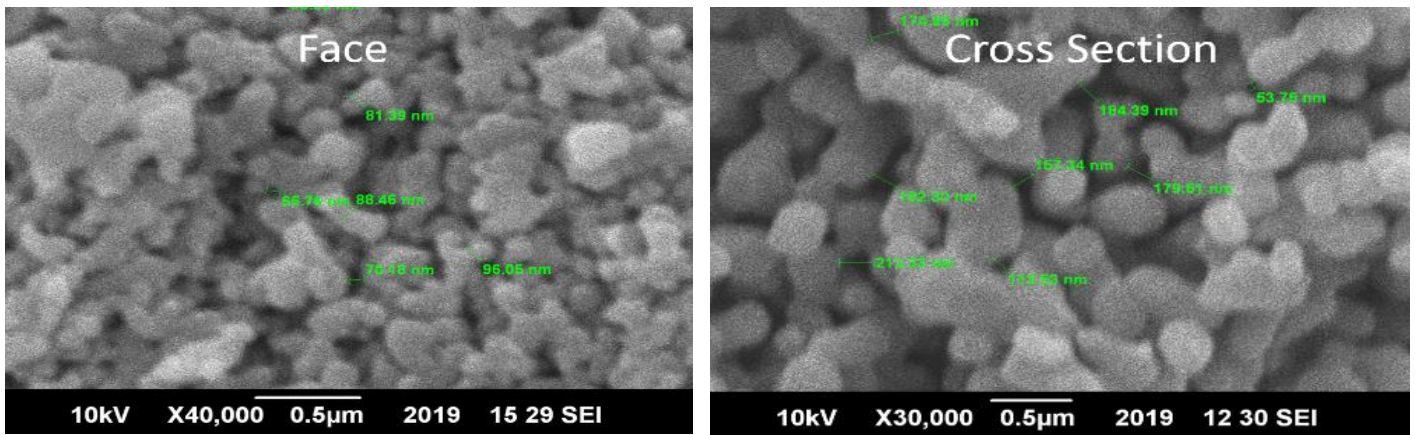
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Figure 27: SEM of 1250°C 15 hours Sample (left: face Right: Cross section)

All samples show a consistent gradual increase in pore size from the face towards the center of the sinter with the necessary open porosity for filtration. The optimum results of pore size were observed in the pellets sintered at 1250°C for 15 hours with an average pore size of 73.6 nm and 119.9 at the surface and cross-section respectively as can be seen in the graph below.
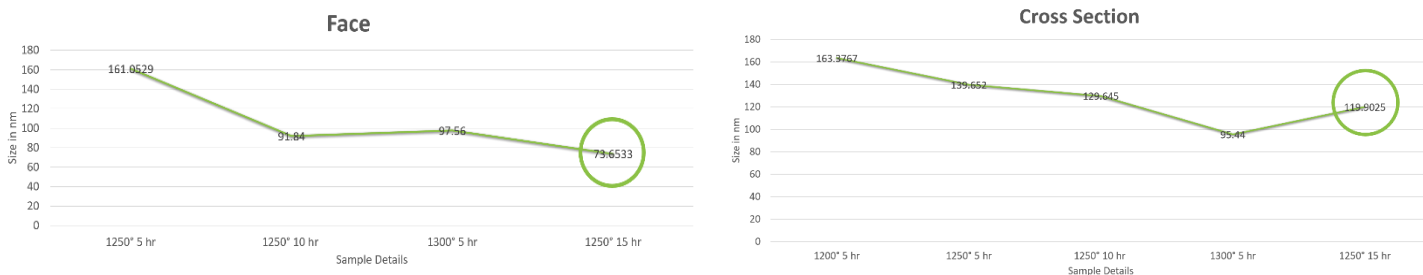

Figure 28: Graphs show the pore size comparison between the samples

4.1.2 Micro Vickers Hardness

Sintered samples were prepared for Micro Vickers hardness testing of each sintering condition; to prepare the samples diamond paste was used to polish the surface so that it becomes easier to measure the hardness under the microscope of the apparatus. The scale set for the microhardness test was Hv_1. The average sample diameter of the sintered products was 2.54 cm, with average thickness 0.2 cm. The sintering parameters for the

samples: 1200°C for 5 hours; 1250°C for 5 hours; 1250°C for 10 hours; 1250°C for 15 hours; and 1300°C for 5 hours. The Micro Vickers hardness values given in the figure are of the average values of each sintering condition. The highest microhardness achieved was 158.3, of samples sintered at 1300°C for 5 hours. Although this sintering cycle results in a sinter that exhibits superior hardness, it compromises increased brittleness on higher pressures and lower porosity and was therefore discarded. The optimized sample has a Micro Vickers hardness of 109.7.
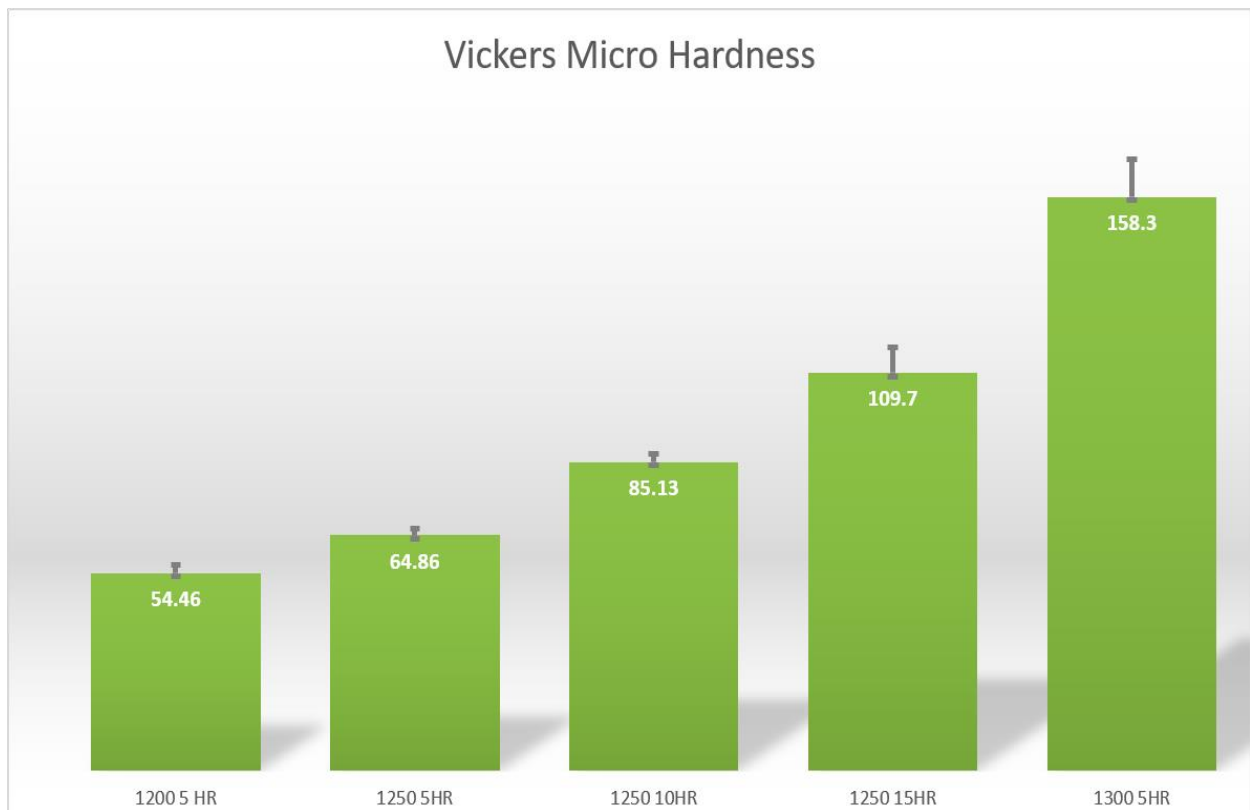

Figure 29: Graphs shows average hardness of the samples

4.1.3: Compression Testing

Similar procedure was followed, and compression testing was performed on Shimadzu Tensile Testing machine with a strain rate of 0.050 mm per minute applied on each sintered sample. The results are shown below:
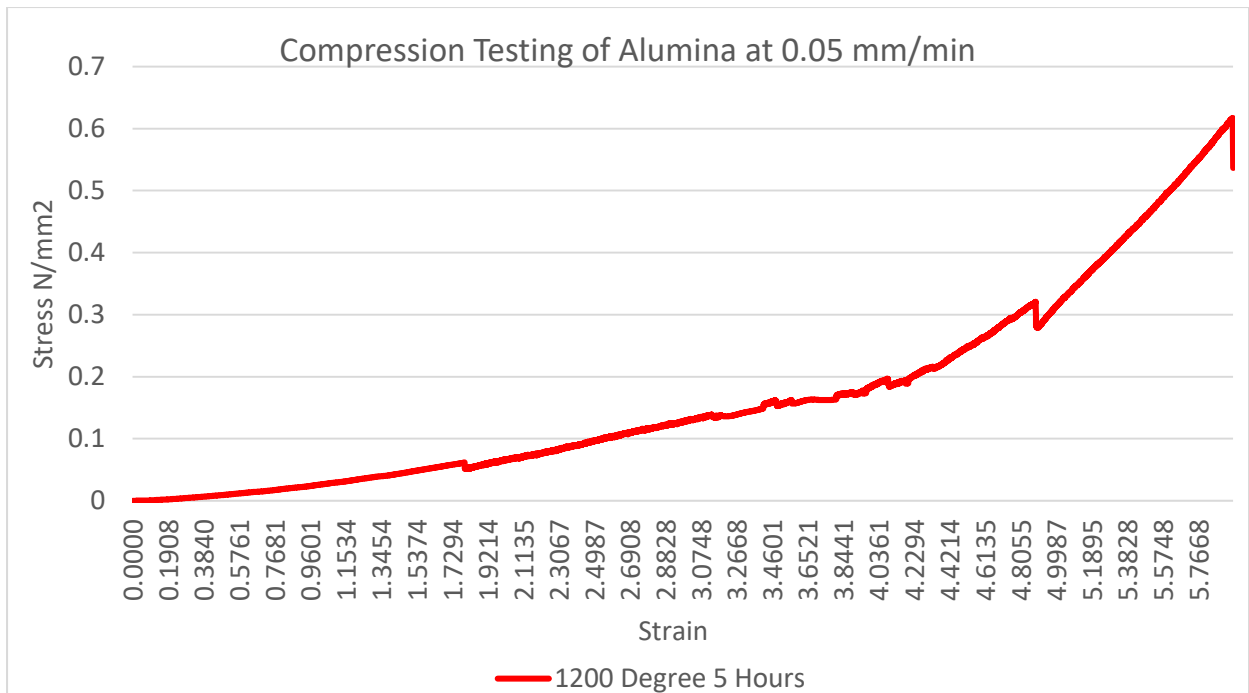
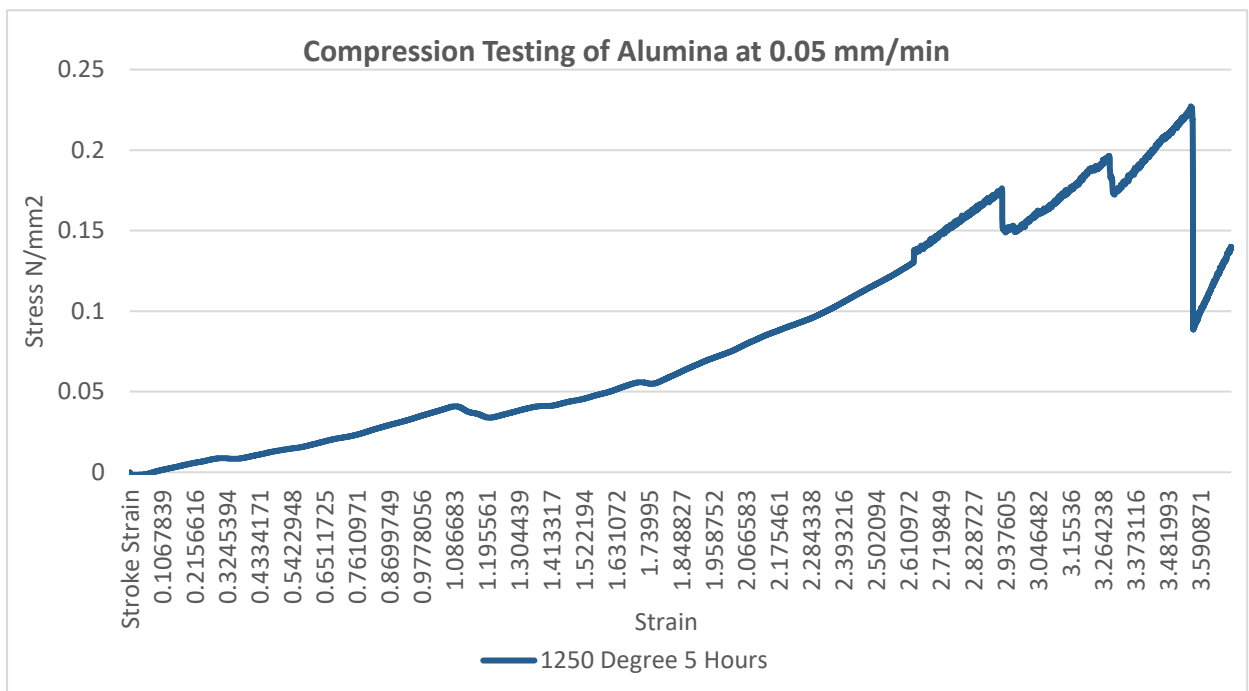Figure 30: Compression Test Result of sample sintered at 1200°C for 5 Hours

Figure 31: Compression Test Result of sample sintered at 1250°C for 5 Hours
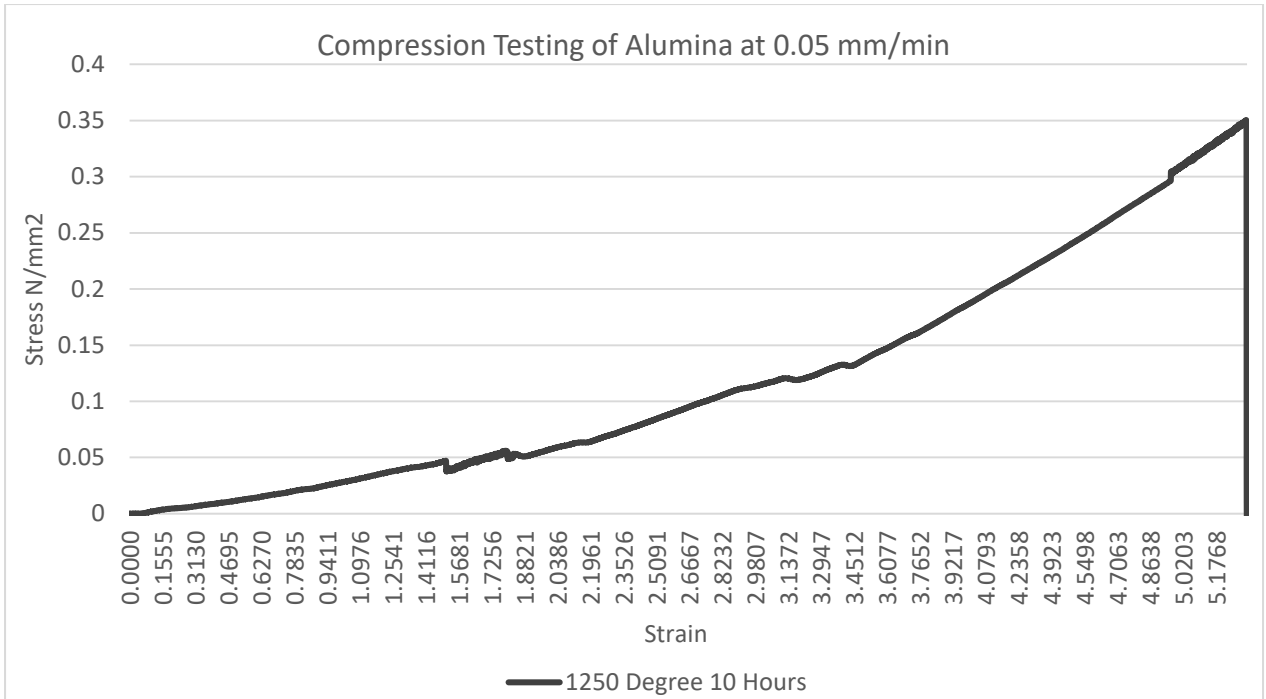
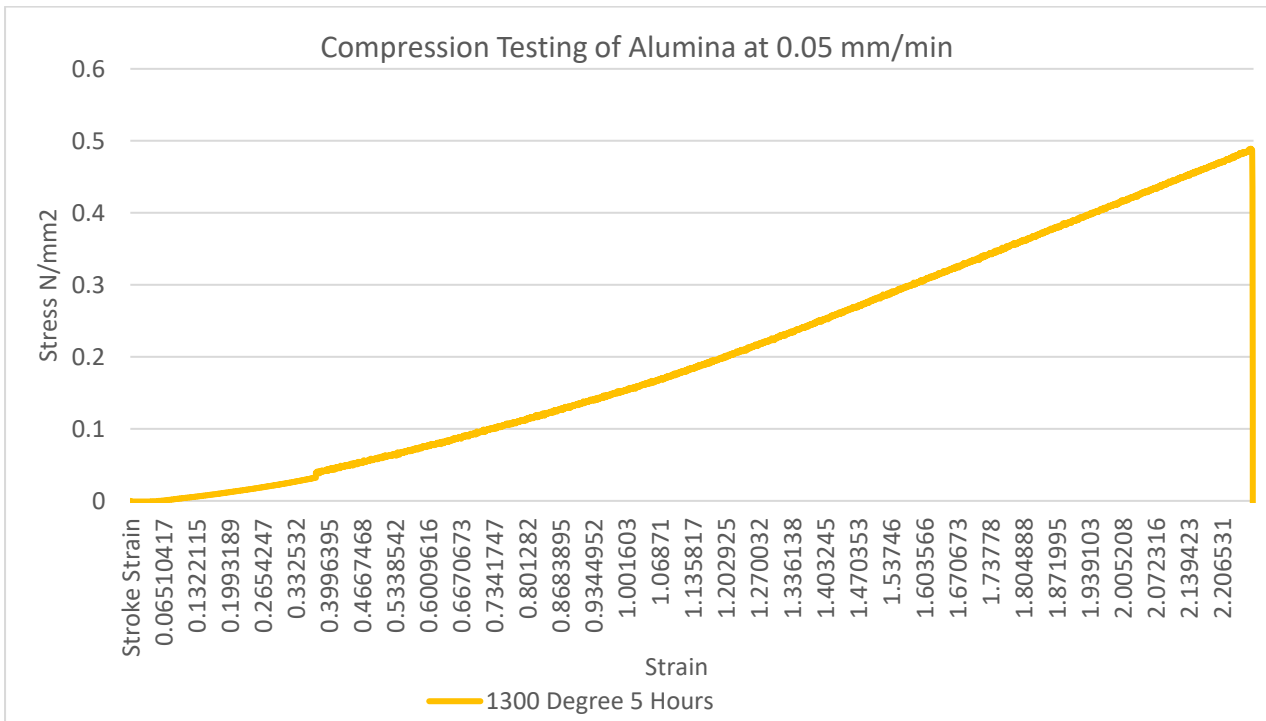Figure 32: Compression Test Result of sample sintered at 1250°C for 10 Hours

X`

Figure 33: Compression Test Result of sample sintered at 1300°C for 5 Hours
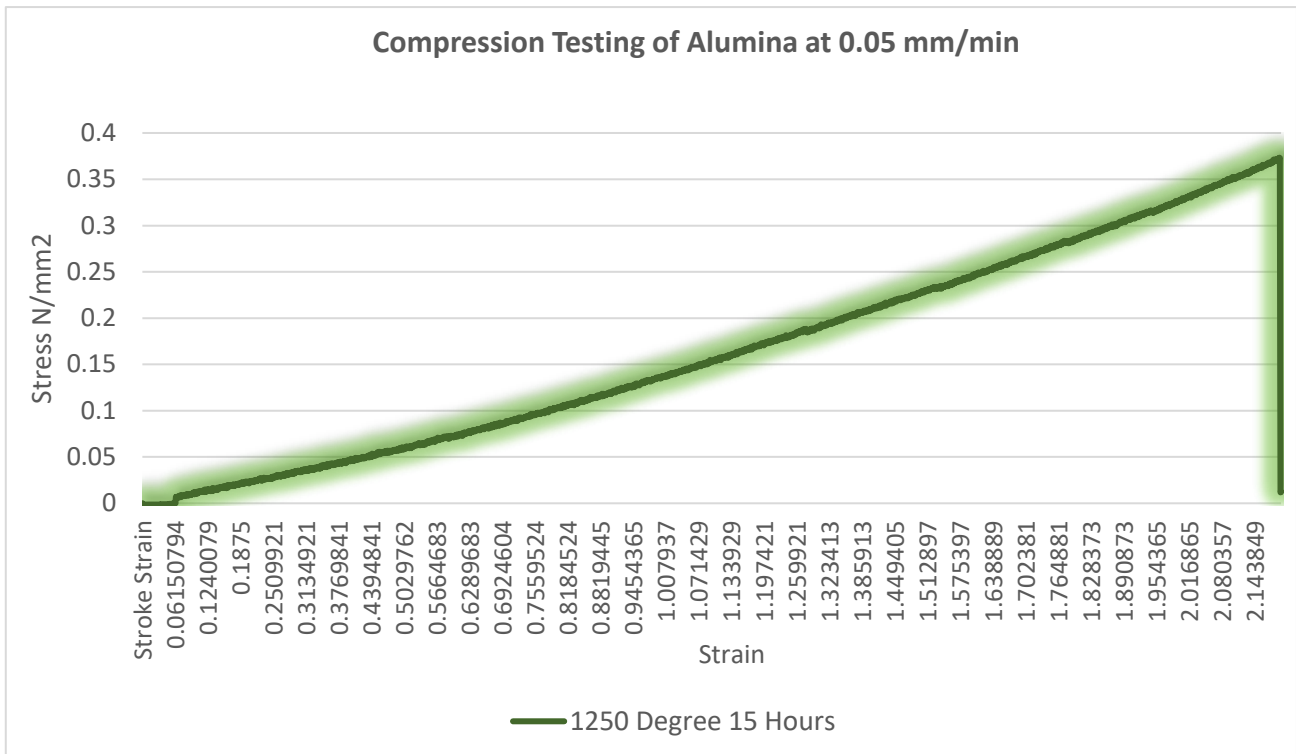
Figure 34: Compression Test Result of sample sintered at 1250°C for 15 Hours

Compression testing of all samples was done to analyze what conditions offered optimum strength in high-pressure working conditions. The reason of carrying out preliminary compression and hardness testing was to simulate the fluid pressures experienced during filtration. It has been later discussed that during lab-scale flow-rate testing of samples, all sintered products could undergo 8–9 bars of pressure.

The constraints presented were firstly to limit pressure to design pressure levels (8 –9 bars) to ensure safety at a lab-scale; and secondly to increase sintering conditions (temperature and sintering time), and therefore the mechanical properties of each sintered sample while keeping in mind the proportional increase in brittleness and reduction in porosity (the number and size of pores decreased, disturbing the open network) with an increase in hardness.

4.1.4 Time-Motion Study

Fresh samples were used of the optimized pellet to perform three tests in each scenario of conditions and average was taken. The time motion graph was made for comparison, as shown in the time-motion study below:

The inlet solutions used were sugarcane juice, distilled water, tap water and muddy water from a flowerpot. All tests have been performed with a fluid pressure of 5 bars against the time measured of each ml of filtrate collected. A large difference can be seen between sugarcane juice and water samples. This is because of the high viscosity of the juice requiring higher pressure to pass through the pores (testing at higher pressures than 5 bars for this experiment was not carried out due to safety concerns). This data was used to calculate the flow rate of the fluids.
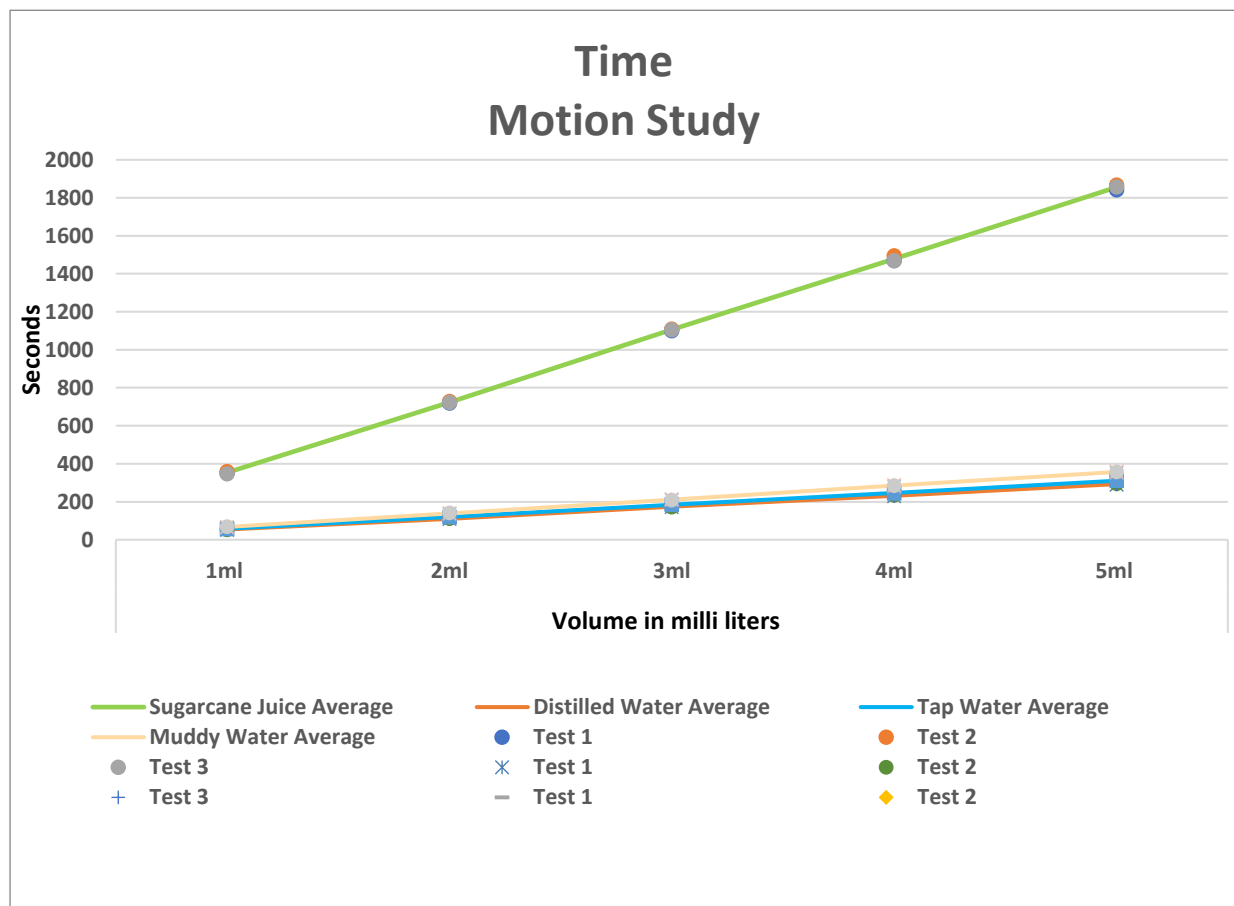

Figure 35: Graph illustrate the time needed for different liquids to flow through the pellet

4.1.5 Flow Rate Testing

Using the time motion study data obtained through experimentation flow rate of all the fluids can be calculated as seen in the graph below. The graph shows the flowrate to be very slow for all the liquids passed through any/all the filters that is due to very small surface area (having a radius of only 6.35 mm) of the pellet. If these very pellets were to be made for industrial usage the flowrate would be much higher as the surface area that filters the juice would increase exponentially.
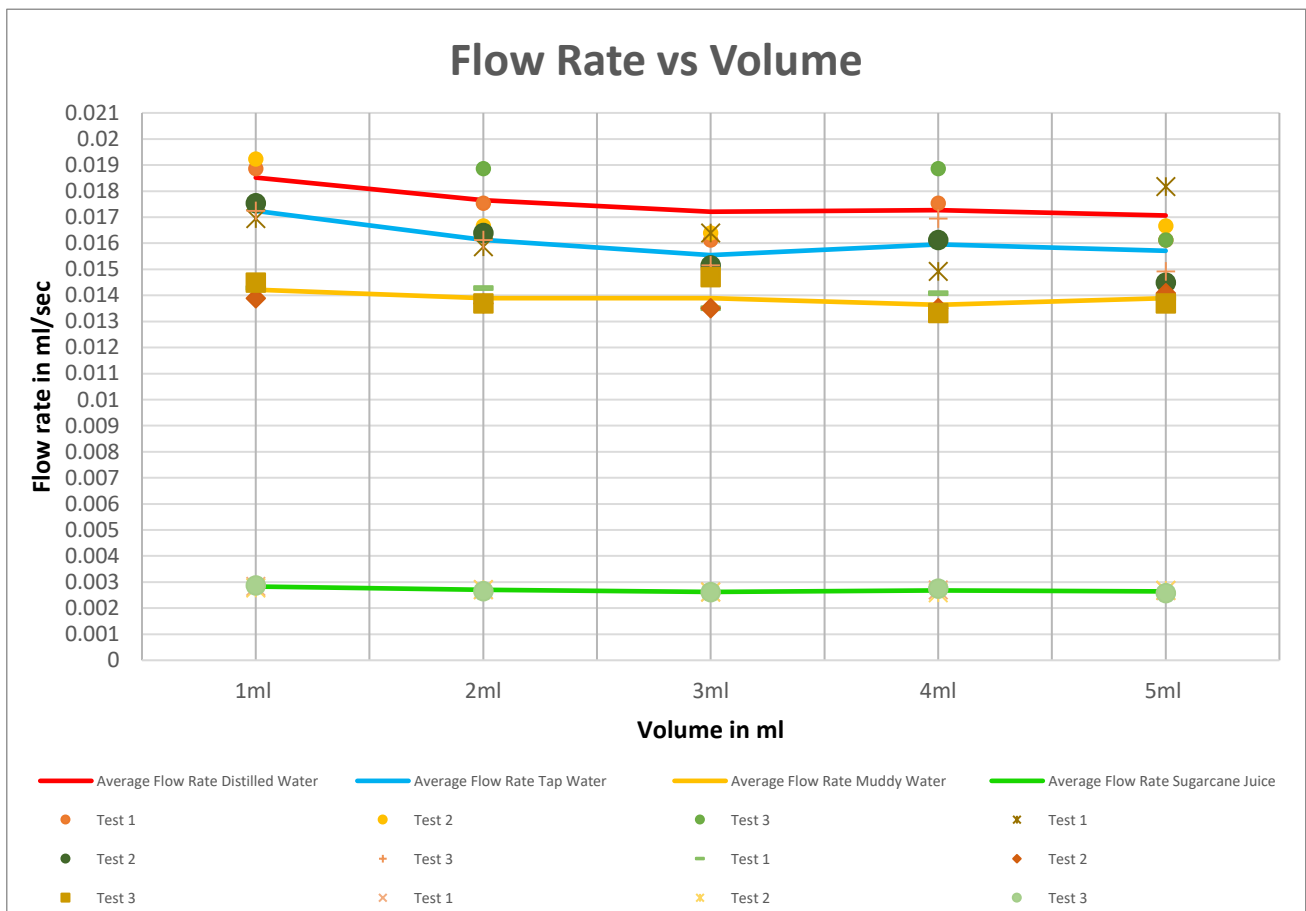

Figure 36: Graphs compares the flowrate of different liquids passing through the pellet

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4.2.0 Filtration Results

Filtration was carried out at a lab scale, keeping pressures between 5–8 bars of pressure. As can be seen below, through a lab-scale filtration process, complete discoloration has been observed as seen by the eye.
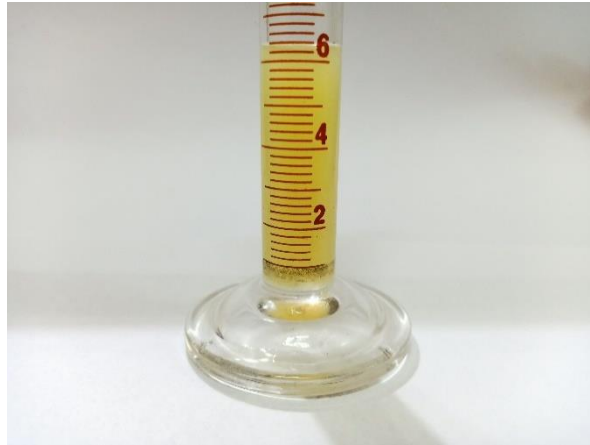
Figure 37: Sugarcane Juice Sample before filtration

Figure 38: Sugarcane Juice Sample after filtration

The pressure levels during this experiment were kept at the design pressure at a lab scale (maximum 9 bars) so as to prevent failure of the apparatus and due to safety concerns; the sintered products can withstand much higher levels of pressure. It was observed that all samples had sufficient mechanical properties through compression and microhardness testing. The porosity resulting from sintering of all samples was observed by SEM to be – at average for all sintering conditions – sufficient to obtain an open network for filtration.

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The sintered product of 1250°C at 15 hours was optimized and flow-rate testing of different fluids showed that at the design pressures of the lab-scale apparatus, a clear relation between pressure, time and volumes can be clarified. In conclusion, discoloration at a lab scale has been achieved using a portable rig setup with minimal power requirements that can be used from the convenience of one's home.

CONCLUSION

In the efforts of making sugarcane refining more cost effective and efficient, the process of ultrafiltration has seen advancements enough to become a more viable alternative, as with time, ultrafiltration technology has become cheaper to produce and implement. The high versatility offered by ceramic membranes with the ability to sustain high-pressure loads, processing anti-fouling capabilities, wear resistance at high temperatures and reverse flow for cleaning cycles so as to prevent anti-fouling; these properties enable them to have a higher operating life compared to polymeric membranes, making ceramic membranes a strong contender for replacing polymeric membranes in the sugar industry. The increase in carbon footprint due to the quicker turnover of polymeric membranes after clogging and/or degradation may be reduced by using ceramic membranes, resulting in a much greener process.

The ceramic membranes created from alumina have showed promising results and offer a good balance between mechanical properties and cost compared to other ceramic alternatives. The testing performed shows that out of several samples created using sintering conditions 1200°C for 5 hours, 1250°C for 5 hours, 1250°C for 10 hours, 1250°C for 15 hours and 1300°C for 5 hours, a sampling procedure has been found with sintering conditions of 1250°C for 15 hours. The optimized membrane exhibits a good compromise between the mechanical strength necessary to withstand high-fluid pressures of up to 9 bars while offering high porosity above a minimum 80 nm and up to 100 nm; the membrane shows a strong interconnected pore network for a smoother filtration process of sugarcane juice and complete discoloration of sugarcane solution has been achieved. Additionally, the ceramic membrane has the capability to be cleaned using reverse pressure flow to dislodge any debris that may be causing clogging, thereby increasing the operating life of the membrane.

The future prospects of an optimized membrane along with a simulated lab-scale apparatus setup for the filtration process are promising; the system, subject to further optimization for industrial usage, may be up-scaled to replace the current sugarcane refinery process at an industrial scale in Pakistan. Locally manufacturing membranes may then be proposed so as to mass-produce ceramic ultrafiltration membranes at an industrial scale, thus economizing the process and reducing the overall operational costs of sugarcane filtration considerably.

In conclusion, the fundamental aims of this research had been to engineer a process that could manufacture an optimized Al_2O_3 membrane – a cleaner product – while also showing promise of making the overall sugarcane refining process greener.

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