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Fabrication Challenges in Synthesizing Porous Ceramic Membrane to Effective Flue Gas Treatment

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ABSTRACT

Global warming is a serious concern worldwide, while there are many contributors to rise the temperature of earth. One major source to it, is air pollution. It is of utmost importance to apply the necessary remedial actions to address the contaminants in outdoor and indoor environment. In this research a step is taken to treat flue gases, for which membrane technology is introduced. A porous ceramic membrane is synthesized from calcined porous alumina (Al_2O_3) and activated washed fly ash. Some other additives like starch $(C_6H_{10}O_5)$ n, binder solution along with ethyl silicate ($C_8H_2O_4Si$) and a deflocculating agent carbonic acid (H_2CO_3), are employed. Alongside it, some of the issues are discussed which are faced during fabrication of porous ceramic membrane i.e., crakes in membrane sample, non-active reactants issue, un-even rise or fall during de-moisturization or sintering steps. Further, the membrane sample is characterized through different test including: Further, the membrane sample is characterized through different test including thermogravimetric analysis (TGA) and DTG, which shown a satisfactory results, as there is negligible percentage weight loss after 750°C. X-ray fluorescence (XRF) for fly ash portrayal and X-ray diffraction (XRD) analysis for structure assessing are conducted, which described that the fabricated membrane has a crystalline structure as like ceramic. Archimedes Principal technique is used to determine bulk density, and porosity of the membrane sample, the values are 4.484 g/cm^3, and 62.5% respectively. Average pore size of 7.6 µm is find out through optical microscopy test, similarly mechanical strength is found to be 2.7 MPa. Furthermore, a pilot scale visual permeability test is performed for flue gases treatment of combusting fuel containing tyre and coal powder. The results show the compatibility of the fabricated porous ceramic membrane to be utilized for treatment of flue gases.

1. Introduction

The rapid growth of the population triggered industrial and infrastructure development. In response, it caused many environmental catastrophes, including global warming, air pollution, water crisis, and waste disposal issues [1]. WHO statistics show that almost all (99%) of the population breathes unhealthy air, contaminated with various levels of pollutants, while exposure in low- and middle-income countries is much higher [2]. Pakistan is ranked 3rd among 118 countries with the worst air quality in 2021, with an average US air quality index (US AQI) of 156. Most Pakistani cities are among the worst polluted cities in the world due to air contamination with PM2.5 and PM10, having average yearly readings of 65.81 µg/m3

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[3]. Specifically, Peshawar is ranked 6th among all cities in Pakistan in 2019 and 37th worldwide with an average particulate matter concentration of 63.9 μg/m^3 yearly. The sources of pollution are emissions from transportand factories including brick kilns, using tyres, wood, and coal for fuel purposes [3]. The ir pollution imposes various environmental and health problems, i.e., global warming, less production of crops, reduction in animal reproduction, and diseases like respiratory ailments with chronic obstructive pulmonary diseases (COPD), skin irritation, and cancer [3]. Most alarming is the death ratio; according to a WHO report, there are 4.2 million deaths every year that occur as a result of exposure to contaminated ambient air [2]. These statistics force researchers and different firms to cope with the situation and reduce future threats to the environment and human lives.

Many techniques are in use to control air pollution which are energy-efficient and optimized process too [4], [5]. Among them ceramic membrane technology is considered the most common, reliable, and effective control technique, with inbuilt features including, a lesser thermal expansion coefficient, high-level corrosion stability, high mechanical strength, enough specific surface area provision, excellent permeability, and light weight compare to metals [6]. To have a maximum transport rate, porous membrane layers must be synthesized with thin layers [7].

At present, many commercial porous ceramics materials are used for the fabrication of membranes including alumina, zirconia, silica, etc. [8], [9] Among them, alumina is considered the most widely used porous material due to its high chemical and mechanical stability and good structure [1]. Sintering difficulty increases due to these oxides' high-level lattice energy, which forces them to have a sintering temperature of more than 1500 ˚C, and pure powdered oxides are expensive [10]. For this reason, to reduce the fabrication cost of porous ceramic membranes and have comparable mechanical and chemical properties, the researcher is working on by-products or wastes, including fly ash. Where fly ash mainly consist of alumina and silica [11]. It has been widely used to prepare ceramic membranes, such as porous flat ceramic membranes synthesized with dolomite[12] and highly porous whisker-structured mullite ceramic membranes [13]. To enhance the properties, different additives are added to the base material, like starch, which is used as a binder and a consolidator or pore-forming agent;carbonic acid, which is an anti-flocculent agent; and ethyl silicate, which is as a binder [14], [15]. There were many applications of polymeric and metallic membranes for CO_2 and H_2S capture[16] and water recovery from flue gas [17]. Moreover, there are not much wide-ranging studies on porous ceramic membranes made of alumina and fly ash mixtures for flue gas treatment. Therefore, this study is conducted to fill the gap.

The aim of this work is to fabricate a porous ceramic membrane, discuss membrane's characterizations, and application of the synthesized membrane for flue gas treatment. The problems that have been mentioned are faced during the membrane fabrication. Supports have been made from fly ash and alumina in order to reduce membrane costs while increasing the mechanical strength and uniform pore structure. These materials were chosen because of their thermal stability and availability. Therefore, the purpose of this study is to create high-quality and reasonably priced ceramic membrane supports by using the physical and chemical features of the base material. The fabricated membrane is utilized on a pilot scale for flue gas treatment.

2. Methodology

2.1 Starting materials

As base materials, alumina and fly ash were selected for the fabrication of the ceramic membrane support. The advantages of the fly ash addition will reduce the cost of base material and it contains mostly silica and alumina where porous alumina will react with silica oxide and will produce mullite which will increase the mechanical strength and uniform pore structure [18] The chemical compounds used for the preparation of ceramic membrane are summarized in Table 1.

2.2 Membrane fabrication

To prepare membrane sample, 5g of calcined porous alumina (as base material) is mixed with 5g of activated washed fly ash manually. Also, two solutions were prepared, first was 30vol% solution of starch and then 1M solution of NaOH and Na_2SiO_3. After that, 5 ml of each solution is mixed and ball milled with base material with ratio of 2.3g of base material/ ml solution. The process is shown in Figure.12 drops of carbonic acid and 3 ml of ethyl silicate are added to the base mixture. The sample paste was finally poured into polyethylene mold with 0.06m diameter. After it the sample is covered fully and placed it in water bath at 60°C for 1 hour for a purpose to avoid any uneven shrinkage.

Table 1: The composition of chemicals used for fabrication of porous ceramic membrane

Chemicals	Formula	Purity (%)	Supplier
Ethyle silicate	Na_2SiO_3	99	HAQ Chemicals Peshawar
Sodium Hydroxyde	NaOH	99	HAQ Chemicals Peshawar
Carbonic Acid	H_2CO_3	99	HAQ Chemicals Peshawar
Alumina	Al_2O_3	99	DAEJUNG Chemicals and Metals Co. LTD
Fly Ash	Contains many oxides, determined through XRF analysis	63%SiO_2 22% Al_2O_3	Lakhra Coal-Fired Power Plant, Sindh, Pakistan
Starch	$(C_6H_{10}O_5)_n$	99	DAEJUNG Chemicals and Metals Co. LTD

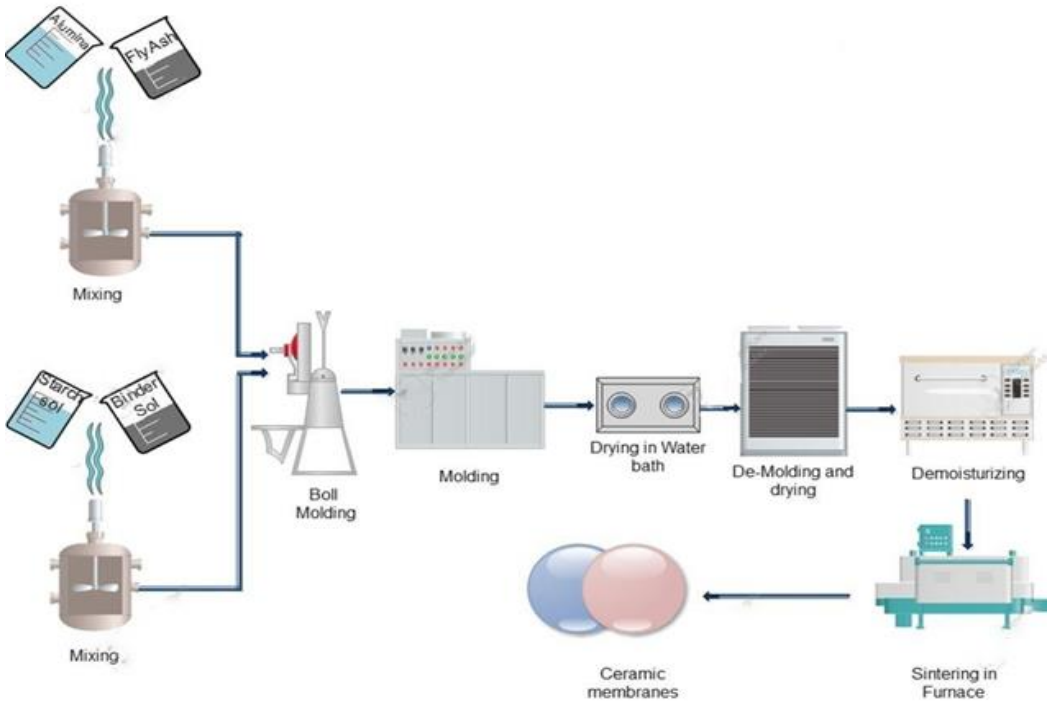

Figure 1. Process flow diagram for synthesis of porous ceramic membrane using porous alumina

Then, the sample was quenched for few mints at 13°C and dried in ambient air at 22°C.At the end the demoulded membrane sample is placed in oven for 20 hours at 105°C for the removal of moisture content. Finally, the sample is then passed through stepwise sintering at 1250°C in furnace. The final prepared porous ceramic membrane can be seen in Figure 2.

Figure 2. Membrane sample top view after sintering at 1250 °C in furnace

2.3 Tackling the fabrication obstacles

Fabrication of membrane needs an extreme attention at each step. If the procedures are not followed as described by the researchers, it will lead to failure at each step and will be hard to achieve the desired results. Some of problems are faced during this research are explained here;

1. If the base material is not properly activated, i.e., the alumina must be calcined and the fly ash must be washed and activated. Further, the starch solution and the binder solution must be made reactive, if the material is not activated properly, it will not lead to membrane sample formation. Upon de-moulding fracture will appear as shown in Figure 3.

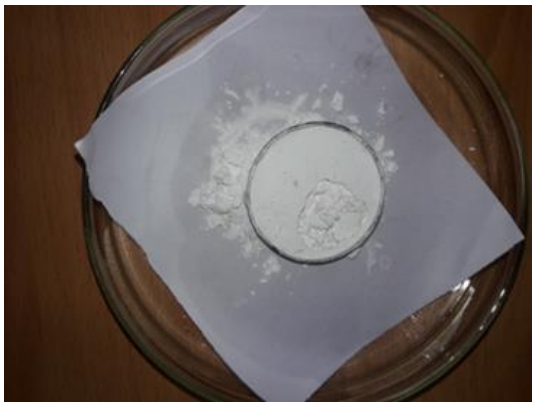

Figure 3. Breakdown of membrane sample upon de-moulding as the alumina was not calcined and starch solution was not prepared properly.

2. Care must be taken during de-moisturizing in the oven; the temperature must be maintained at 105°C for 20h; if there is any sudden fall in temperature it will lead to crakes as shown in Figure 4. It happened due to a stoppage of the power supply to the oven (due to an electrical shutdown in the Lab) when de-moisturization was in progress.

Figure 4. Crack in membrane sample when power supply was stopped to oven during de-moisturization at 105°C for 20 hours.

3. Sintering must be carried out in a stepwise manner, like there must be proper degree rise, i.e., heat rate, then maintaining it for specific time at a specific temperature, and at last reach the required temperature. Any uneven rise or fall in temperature will deform the shape of the membrane sample, as shown in Figure 5.

Figure 5. Swallowing of membrane during sintering, the root cause was uneven rise in temperature during fabrication.

2.4 Characterization methods

The elemental compositions of the principal raw materials, fly ash, are determined by X-ray fluorescence technique at the Material Characterization Laboratory, U.S.-Pakistan

Center for Advanced Studies in Energy, UET Peshawar. For measurement purposes in XRD analysis, the radiation Cu-kα was applied. Where the specimens were fabricated through a backloading mechanism. Whereas a solar 2.5°- and 0.6-mm anti-scatter slit including a diffracted beam is supplied. For the reduction of air scattering, the scattering screen is placed above the sample with a length of 2mm. For the operation of the X-ray tube, the generator position was fixed at 30 kilo volt and 10 mA. Measurements were conducted in a vertical Bragg-Brentano (β-β) geometry in between 10° and 70° for 2Θ angles having a shape size of 0.02° with a rate of 0.6 seconds per step. Thirty minutes is the specific measurement time for each scan. Now coming towards thermal gravimetric analysis, where the paste of the formulation was subjected to thermogravimetric analysis (TGA – SII 6300, Exstar) to identify weight loss patterns. In this test, a powdered membrane sample of 8.5 mg is considered, and for 30 min, nitrogen is passed through the apparatus at a flow rate of 30 ml/min. To remove the volatile organic components, a heat rate of 20 °C/min is fixed until 1250 °C. After it, atmospheric ambient air is introduced. Keeping in consideration the powdered membrane sample must be dried first for two hours at 100°C.

3. Results and discussions

3.1 Bulk density, porosity, and pore size

The bulk density, porosity, and pore size of the synthesized membrane sample were determined at the Material Characterization Lab, Renewable Energy Section, U.S.-Pakistan Center for Advanced Studies in Energy, University of Engineering and Technology Peshawar, to characterize its feasibility. The bulk density of membrane is found to be 4.484 g/cm^3, which is measured through the Archimedes principal technique.

Fly ash ceramic membranes have a theoretical density of 3.16 g/cm^3 and 3.98 g/cm^3, respectively [19]. Only clay was present as the main crystalline phase in the proppants produced for this investigation. Thus, the greater apparent density of the proppants made from fly ash was 4.48 g/cm^3 [20], in contrast to the majority of ceramic proppants in the Chinese market and those produced by Carbo Company, whose density ranges between 2.80 and 3.40 g/cm^3. To sum up, fly ash is used to create high-density ceramic proppants based on the superiority of the single crystalline phase. Furthermore, the percentage porosity for the sintered membrane sample is 62.5% according to Archimedes principle. The average pore size of the membrane is determined using optical microscopy, as shown in Figure 6 with a value of 7.6 µm.

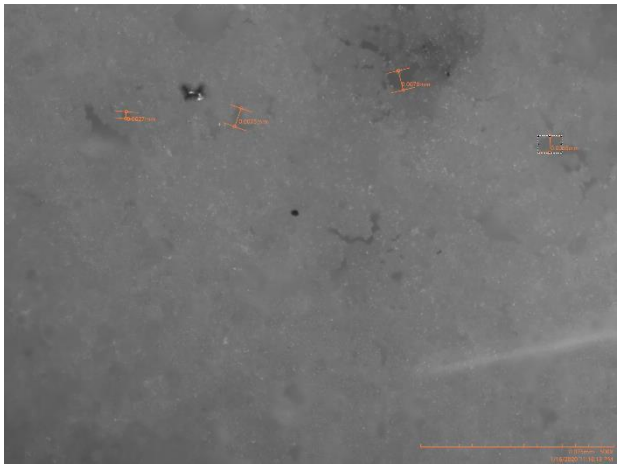

Figure 6. Average pore size of the sintered membrane sample (500X resolution).

3.2 Mechanical strength

The mechanical strength is finding out at UTM Lab, USPCAS-E, UET Peshawar, through a graph of force vs deflection shown in Figure 7. Recorded bending strength value is 2.7 MPa for the sintered membrane sample. The same patent has been mentioned in the literature [21]. The high sintering temperature of 1250℃ resulted in high mechanical strength. This is mostly because of the formation of a neck since the higher sintering temperature gradually eliminates considerable grain expansion or contraction and increases the membrane's strength [22].

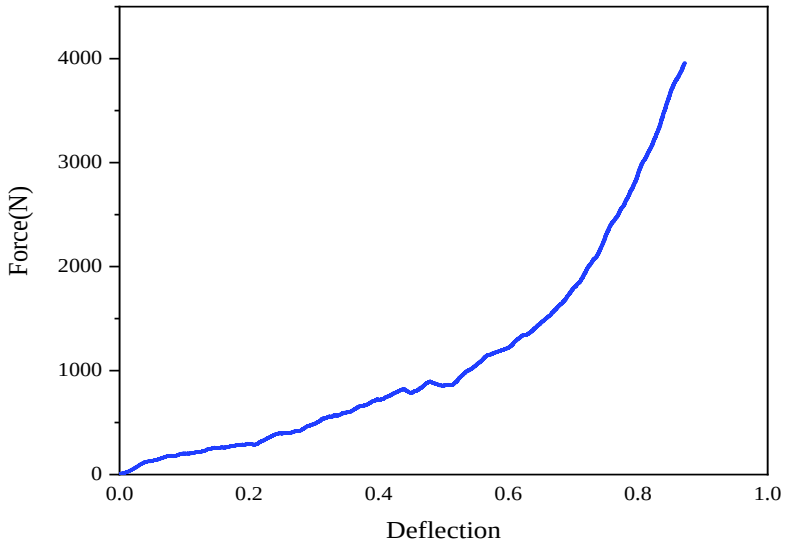

Figure 7. Mechanical strength found 2700 KPa, a fracture is found in the sample at 4000 N force.

3.3 X-ray diffraction (XRD)

X-ray diffraction spectroscopy (XRD) results show the crystallinity and total reactivity of the membrane which is determined at different peaks values showed in Figure 8. Powdered sample has many phases where a lot of constructive interferences can be seen as the peak show in detail in Figure 8. Dehua et al. also obtained close results during studying the effect of sintering temperature on the membrane pattern at different temperatures [23].

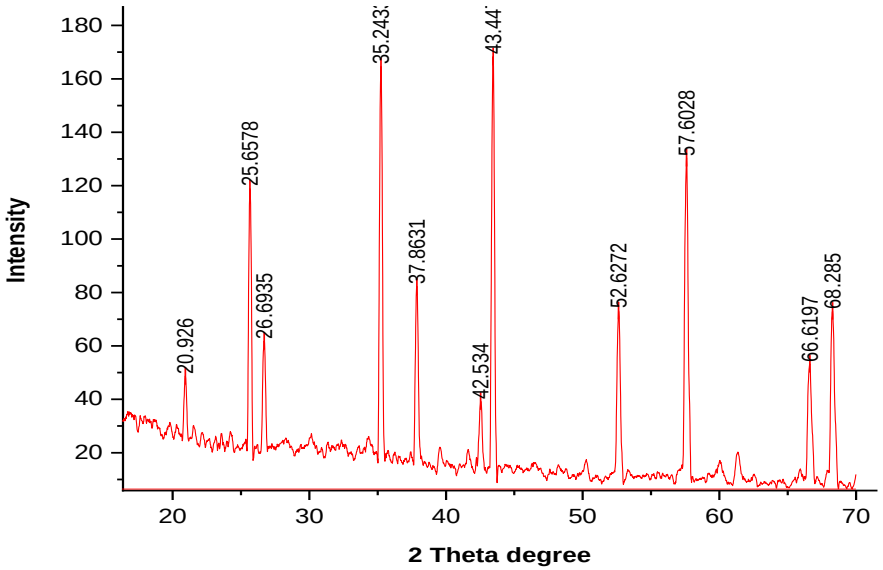

Figure 8. XRD patterns of porous ceramic membrane sintered at temperature of 1250℃.

3.4 Thermogravimetric analysis (TGA)

Thermo gravimetric analysis (TGA) at Material Characterization Laboratory, U.S.-Pakistan Center for Advanced Studies in Energy, UET Peshawar, is performed for the non-sintered membrane sample powder (i.e., it's the slurry powder which contains starch and other volatile oxides and moisture content) where it shown a satisfactory result that the ceramic membrane will sustain at high temperature and should be utilized for the

treatment of exhaust gases. With the increase in temperature, mass loss decreases. And the total mass change resulted to be 28.96%. At the start as the sample is not sintered, due to which the de-moisturization and burning of starch occur, and after that a negligible amount of loss in mass is found in the sample. The percentage weight loss is shown in Figure 9. This weight loss is due to the presence of either moisture content, or starch content which works as a pore forming agent cause during sintering it burns and leave pores behind. Further there are some volatile oxides which can sustain till 750°C. As this TGA analysis is carried out till 1200°C. Again, referring to Figure 9, there is no loss in percentage weight after 750°C which explains that this ceramic membrane is sufficiently thermally stable and is ideal to be used for flue gas treatment.

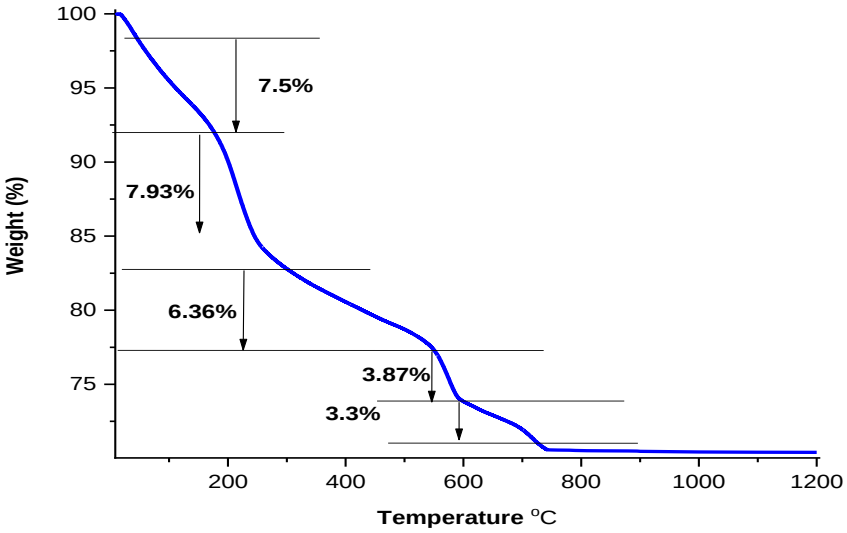

Figure 9. Thermal gravimetric analysis (TGA) result of non-sintered membrane powder

To make the graph easier to comprehend, the first derivative of mass percent vs. temperature can also be displayed. The first derivative is applied to the original picture in order to determine the maximum temperature peak of each mass loss. This is known as inflection point. Figure 10 showed the highest temperatures at which the ceramic membrane loss some weight, are at 212.85°C due to de-moisturization and starch combustion and 576.47°C due to removal of volatile oxides.

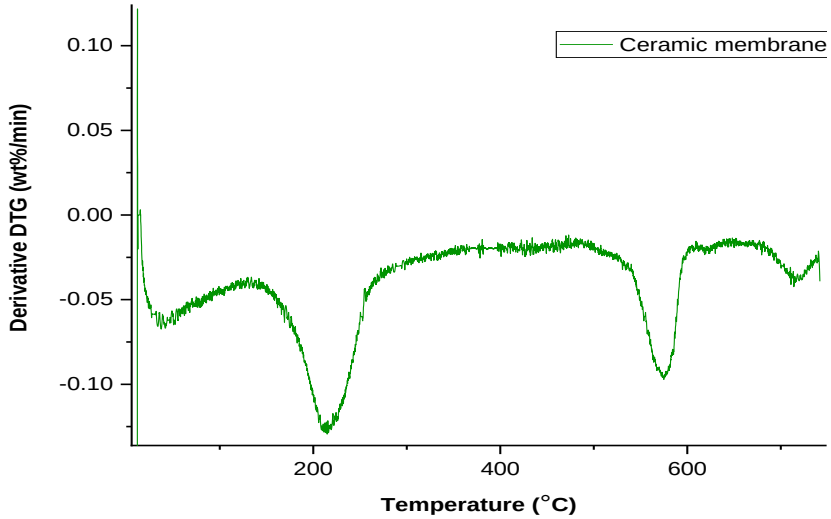

Figure 10. The first derivative DTG of ceramic membrane

4. Visual test

The pilot scale setup contains two parts as shown in Figure 13, one is a combustion chamber with a chimney, while the second part is a glass tube containing the membrane sample and a suction pump at the top end (full description; two-portion glass tube having upper and lower part containing porous ceramic membrane in the middle is utilized. Where lower part is exposed to the flue gases coming out of the combustion chimney. Similarly, the upper portion of the glass tube is connected with a vacuum pump through a tube. The pump creates a vacuum with the suction facility where the exhaust gases of the chimney are induced through the tube-containing membrane in the middle. Further, fuel used for combustion is a mixture of tyre and coal powder. The flue gas goes through the chimney and then passes through the glass tube where the membrane is fixed in the middle by the force of vacuum pressure. The results are very satisfactory where the black smoky particle stuck on the membrane surface which is exposed directly to the chimney. The cake produced and the lower part of the tube shown in Figures 11 and 12 which is comparatively colored more blackish, proposed that it's highly recommendable to utilize a porous ceramic membrane for flue gases treatment.

Figure 11. The Cake of black smoky particle produced at the surface of membrane exposed to chimney flue gases passage, the test took 2 mints.

Figure 12. Shows the glass tube where the bottom end is directly connected with chimney, and upper end is connected with vacuum pump. The membrane is placed at the middle of glass tube. Upper part of the glass tube is less blackish compare to lower part as the flue gas is filtered to some extent.

Figure 14. Pilot scale visual permeability test experimental setup

5. Conclusions

This research article highlights the importance of addressing air pollution as a major contributor to global warming. The study focuses on the treatment of flue gases using a porous ceramic membrane synthesized from calcined Al_2O_3 and activated washed fly ash, along with other additives. The fabrication process of the membrane encountered some challenges such as cracks, non-active reactants, and uneven rise or fall during de-moisturization or sintering steps. However, the membrane

sample exhibited satisfactory results in various characterization tests, including thermo gravimetric analysis (TGA) and DTG, which showed negligible weight loss after 750°C. The X-ray fluorescence (XRF) and X-ray diffraction (XRD) analyses confirmed the ceramic-like crystalline structure of the fabricated membrane. The membrane demonstrated a bulk density of 4.484 g/cm3, a porosity of 62.5%, an average pore size of 7.6 µm, and a mechanical strength of 2.7 MPa. A pilot scale visual permeability test conducted for flue gases treatment involving combusting fuel containing tyre and coal powder showed the compatibility of the fabricated porous ceramic membrane for such applications. Overall, these findings indicate the potential of membrane technology for addressing air pollution and treating flue gases, contributing to efforts in combating global warming.

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