Fabrication and Characterization of ceramic membrane by Gel Cast technique for water filtration

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Abstract: Sayong ball clay membrane can be fabricated without the assistance of high-tech sophisticated machineries and complicated production methods make the filters particularly attractive as a point-of-use treatment. This work was concerned with the study of Sayong ball clay as a wastewater filtration membrane by Gel Cast technique. Gel cast involves mixing process of Sayong ball clay with the solvent, monomer, dispersant, initiator and catalyst in a certain composition. Tubular membrane was designed and fabricated by controlling the Gel Cast monomer (5, 10, 15, 20 wt% of MAM). The physical measurements (shrinkage, apparent porosity, bulk density), microstructure analysis, filtration process (flow rate) and water quality assessments (pH, color, COD, SS) were carried out at different wt% of MAM. The experimental results showed that the apparent porosity and bulk density of Sayong Ball Clay produced by Gel Cast with 5 wt% MAM were 15.39% and 1.87 g/cm^3, respectively. It gives improvement in water quality with the lowest suspended solid (192 mg/L), lowest COD (4 mg/L) and most colorless (1.1 Gardner units) filtered wastewater as compared to others. For microstructure, the porosity increases with increasing MAM wt%. Changes in MAM wt% are likely to have some effect in filtration process as increases the MAM wt% will increases the flow rate of filtered wastewater due to the higher porosity.

1. Introduction

Gel Cast was developed to overcome some of the limitations of other complex-shape forming techniques such as injection molding and slip casting. In Gel Cast, concentrated slurry of ceramic powder in a solution of organic monomer is poured into a mold and then polymerized in situ to form a green body in the shape of the mold cavity. It is a combination of polymer chemistry with slip technique and represents minimal departure from standard ceramic processing. The simplicity of the process has attracted industrial partners and by collaboration between them and the developers, the process is being advanced from the laboratory toward industrial production [1]. Filtration is commonly the mechanical or physical operation which is used for the separation of solids from fluids (liquids or gases) by interposing a medium through which only the fluid can pass [2]. Oversize solids in the fluid are retained, but the separation is not complete; solids will be contaminated with some fluid and filtrate will contain fine particles (depending on the pore number)[3]. The hollow fiber membrane filtration modules were selected as they offered substantially high packing density around 9000 m^-2 m^-3 as compared to packing density of around 30-500 m^2 m^-3 offered by the plate and frame or tubular membrane format[4]. The importance of filtration technologies for waste water treatment has escalated in recent years. In contrast to the conventional, ceramic matrix composites have high mechanical, chemical and thermal endurance as they can be regenerated and there is no ageing process of the material [5]. Ball Clay (Sayong Ball Clay or Bercham White Ball Clay) are Kaolinitic sedimentary clays that commonly consist of 20-80% Kaolinite, 10-25% mica, 6-65% quartz. Localized seams in the same deposit have variations in composition, including the quantity of the major minerals, accessory minerals and carbonaceous materials such as lignite. They are fine-grained and plastic in nature. Testing of water treatment is important in identifying a filtration system is efficient and functioning correctly according to the desired application. Additionally, results of the test also indicate whether the water fulfils a quality
of standard water [6]. Water quality is the physical, chemical and biological characteristics of water. It is a measure of the condition of water relative to the requirements of one or more biotic species and or to any human need or purpose.

2. Experimental procedure

2.1. Materials

The following materials were used in this study. (1) Sayong Ball Clay: particle sizes ≤100µm and the chemical composition of Sayong Ball Clay is given in Table 1 (Techno Ceramic Sdn. Bhd), (2) Methacrylamide/MAM, (3) N,N'-Methylenebisacrylamide /MAD, (4) 1-Octanol, (5) Ammonium Peroxodisulfate/APS, (6) Tetramethylethlenediamine/ TEMED.

2.2. Methodology

The MAM solution (5, 10, 15, 20 wt% of MAM) prepared and mixed with MAD powder in the ratio of the solution to the MAD powder was 6:1. The mixture then mixed with Sayong Ball clay (55% of the total mixture) for 24 hours. The second mixing continued for another 3 hours by adding 0.1% of 1-Octanol, 0.1% of APS solution (10 wt%) and 0.05% of TEMED. To obtain homogenous slurry, the mixing progress was running on a roll milling machine under 153.8 revolutions per minutes [7] with added zirconia balls (1.3(powder): 1(zirconia ball)) to assists the mixing process. The mixture then been cast into the prepared mold and left in high humidity (at 25°C) for 2 hours before undergo drying progress by using oven at 60°C for 30 minutes before unmold. The green casts were pre-sintered for one hour at 600°C to burn out MAM before sintered at 1300°C [8]. The rate of heating and cooling was 10°C per minutes and soaking time was 20 minutes for every green cast. Initial and final dimensions were measured before and after sintering process for shrinkage measurement. The bulk density and apparent porosity of the samples was measured by the Archimedes method using distilled water. The microstructures were observed by FESEM. The tubular membrane was attached to the wastewater filtering system and its flow rate was measured. The outputs of wastewater were tested for pH, Chemical Oxygen Demand (COD), Suspended Solid (SS) and color.

Table 1: Chemicals composition (%) of Sayong Ball Clay

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<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>TiO₂</th>
<th>CaO</th>
<th>MgO</th>
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<td>0.14</td>
<td>0.17</td>
<td>13.97</td>
</tr>
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</table>

3. Results and discussion

3.1. Physical measurement and filtration process.

Fig. 1 summarizes the shrinkage and bulk density of samples for different MAM wt%. The bulk density of samples was in the range from 1.36 to 1.87 gcm⁻³. The phenomenon of shrinkage occurs as collapse of the 3D network structures of cross-linked polymer gels [9]. The contents of monomer and cross-linker in slurry influence the 3D network structures of cross linked polymer gels. As expected, a high MAM wt% leads to a decrease of bulk density. The difference in bulk density for difference MAM wt% can be related to their apparent porosity and microstructures.
Fig. 1: Shrinkage and bulk density of samples for different MAM wt%.

Fig. 2 shows the apparent porosity and flow rate of samples for different MAM wt% sintered at 1300°C. The reduction of flow rate can be explained by the apparent porosity which will reduce the efficiency of membrane and the pores mainly originated from the residual micro space [10] of the organic monomer (MAM) in the green body during organic binder burn out. Increasing MAM wt% (5-20 wt%) will increase the apparent porosity and flow rate.

3.2. Microstructure.

The effect of MAM wt% on the microstructure was shown from the microstructure of Gel Cast sample (Fig. 3). The microstructure indicates that the powders in green body distribute compactly and also the grains are connected by polymeric networks [11], which contribute to distribution of porosity in the samples. The sample with highest MAM wt% (20 wt%) shows the most possesses porosity as compared to sample with lowest MAM wt% (5 wt%). This occurs as the MAM wt% increases will result increases of the MAM cross linking rate in the polymerization and reduce the probability of inhibition of the cross linker that lead to collapsing of polymer network, thus increasing the porosity in the sample. Monomer and cross-linker herein not to hold the ceramic particles together, but also play a leading role in pore formation [12].
3.3. Water Quality Assessments.

The pH values for filtered and unfiltered wastewater show in the range of alkaline. MAM wt% not gives effect in pH value. From the data of suspended solid testing (SS) for unfiltered wastewater content highest suspended solid that was 81948 mg for every 1 liter compared to filtrated wastewater. This shows that tubular ceramic membrane can reduce suspended solid in unfiltered wastewater. In term of MAM wt%, as increase the wt% will increases the suspended solid in one liter of wastewater by 70% from sample sintered at 5 wt% to 20 wt% MAM. Data of COD testing shows that unfiltered wastewater gives highest COD value that is 299 mg of oxygen mass for every 1 liter compared to overall filtered wastewater. This results indicates that organic pollutant in unfiltered wastewater was highest compared to filtered wastewater. This can be observed from the color and precipitation in unfiltered wastewater as existent of the organic pollutant compare to colorless filtered wastewater. Also increases the MAM wt% will increases the value of oxygen consumed per liter of wastewater from the wastewater filtered by sample with 5 wt% to wastewater filtered by sample with 20 wt% MAM. Wastewater filtered by sample via Gel Cast method shows lower in its’ true color compare to unfiltered wastewater. This indicates that color intensity in unfiltered wastewater is darker (305 Gardner units) than color intensity in wastewaters via sample produced by Gel Cast method. The MAM wt% gives influence as true color between wastewater produced by sample with 20 wt% MAM was highest (3.1 Gardner units) compare to wastewater produced by sample with 5 wt% MAM (1.1 Gardner units). Suspended solid can increases levels of dissolved oxygen (COD) as reported by Appleby and Bloesch [13]. A decrease in water clarity (color) is another obvious change resulting from an increase of suspended solids.
4. Conclusion

The Gel Cast sample with lowest MAM wt% (5 wt%) shows minimum apparent porosity (15.39%) and maximum bulk density (1.87 g/cm³) and shrinkage (20.58%). Based on microstructure observation, the porosity increases with increasing MAM wt%. Changes in MAM wt% are likely to have some effect in filtration process as increases the MAM wt% will increases the flow rate of filtered wastewater due to the higher porosity (higher rate of cross links in polymerization mechanism). Overall, for wastewater filtered by Gel cast sample with 5 wt% MAM show improved in water quality (lowest COD: 4 mg/L and most colorless: 1.1 Gardner units).

References