# Characterization of Local Mineral Materials for Geopolymerization Technique

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## Abstract

Subject matter of this study is characterization of mineral material used for geopolymerization techniques. Source mineral material for geopolymerization was collected from Matani Azakhel, which is situated 32 km from Peshawar, Khyber-Pakhtunkhwa Pakistan. Selected source material was appropriate due to the available ratio of silica (Si) to alumina (Al) i.e. 3.01, required for geopolymer gel preparation. Thermal activation at 700 °C convert the mineral material into Metakaolinite. Conversion of Si and Al source material into metakaolin were characterized by XRF, XRD and FTIR. The help of sodium hydroxide and sodium silicate with the ratio of 4.3% did chemical activation of source material. Geopolymeric gel was prepared $Na_2O$–$3SiO_2$–$Al_2O_3$ in moulded in stainless steel with the diameter of 10 mm by mixing of metakaolin and Chemical activator. The hydrothermal treatment of molded paste was carried out at 90 °C for 15 hours. Pore diameter of synthesized membrane was examined before and after sintering temperature through scanning electron microscopy. An average pore diameter of 1.0 to 1.3 µm was found before sintering temperature that reduced to an average diameter of 0.6 to 0.8 µm. Universal testing machine show pine cracks at 62 MPa, which shows high compressive synthesis of ceramic membrane.

**Keywords:** Mineral material, XRF, XRD, FTIR, TGA, SEM, Geopolymerization.

### 1. Introduction

Davidovits in 1978, introduced the term geopolymer as a new binder material with amorphous microstructure used in geopolymerization technique for membrane preparation [1]. Geopolymerization is a fast heterogeneous chemical reaction of solid aluminum silicate with other highly concentrated materials like sodium or potassium hydroxide and saturated silicate solution that produces a solid compact material of aluminosilicate which is called geopolymeric material. Geopolymerization technique is stepwise that totally depends on source material and alkaline liquid, for chemical activation [2]. In this two-step mechanism process, thermal activation of source materials is carried out first and chemical activation of activated sources materials were processed through normal or separate mixing [3].

Geopolymeric source material is selected on the basis of aluminum (Al) and silicate (Si) contents presents. Such types of materials are naturally available in the form of mineral kaolinite, red mud and sand clay [4].

Si and Al in the source materials are bonded together with the help of a geopolymeric alkaline liquid that acts as a binder to synthesize these microstructure materials. Alkaline activation of source material is referred to the chemical reaction that either partially or fully changes the microstructure of the source materials [5].

Chemical activators in geopolymerization may sodium hydroxide (NaOH) or potassium hydroxide (KOH) and Sodium silicate ($Na_2SiO_3$) or Potassium hydroxide ($K_2SiO_3$). When metakaolin come in contact with Sodium hydroxide, leaching of Si and Al started with other ions during mixing [5-6].

Geopolymerization technique is very environmentally friendly technique because it uses industrial residues, waste products and sub-products as raw material. Therefore technology of geopolymerization is called *Green Technology* [7]. Also geopolymeric membrane has excellent and admirable compressive strength, inertness to aggressive and harsh environmental and high engineering performance as compare to other conventional membrane technologies.

The term membrane separation is one of the most economical, sustainable and environmental friendly separation process as compare to other conventional separation processes like leaching, extraction distillation and absorption. Membrane separation is cost effective because no phase change involve during separation [8].The term membrane is a barrier between the head to head phases under driving force of pressure, electric field and concentration [9].

Development in membrane operation in the last decades replaces many existing industrial processes and reduced energy consumption. There are two types' membranes, *(i)* organic and *(ii)* inorganic. Both type of membranes are used in water treatment, gas separation and other various industrial applications [10]. Organic membrane have low resistance to harsh chemical environment, high temperature and pressure, while Inorganic membrane show long term stability, inertness to microbial elements and excellent resistant to harsh environment. Also inorganic membrane can be used as a membrane reactor in different operation where both separation and reaction is necessary [11].

Inorganic ceramic membranes are normally composed of silica, titanium and aluminum oxides and metals ions [12]. The specific properties of inorganic ceramic membranes motivate researcher because such material are cost effective and environmentally friendly. Excellent stability of inorganic ceramic membrane makes it suitable for use in food, pharmaceutical and other operational industries for microfiltration and ultrafiltration [13].

In the current study, we attempt geopolymerization techniques for inorganic ceramic membrane synthesis in round flat shape using locally available mineral material that was collected from Matani Azekhel Peshawar KPK, Pakistan. Inorganic ceramic microfiltration membrane was characterized by XRD, FTIR, SEM and Universal testing machine. We have also investigated the effect of percent ratio of chemical activator ($Na_2SiO_3$/NaOH) on compressive strength of synthesized membrane. Surface characterization of the inorganic ceramic membrane prepared through geopolymerization technique will help us in identifying the local source material suitability for membrane preparation. Also this analysis will show that prepared membrane is defect free and process of preparation is cost effective as compared to other imported membranes available in the market.

## 2. MATERIAL AND EXPERIMENTAL PROCEDURE

### *2.1 PREPARATION OF SILICA AND ALUMINUM SOURCE MATERIAL AND ALKALI'S ACTIVATORS:*

Silica and aluminum source materials were collected locally from Mattani area, 25km south of Peshawar, Pakistan as shown in Figure 1. Dumped of source material were spread over the area of 200 $km^2$ of Mattani Azakhel, 9 to 20 feet below the surface of the earth. Source material from different points in the same area was collected and mixed for compositional characterization. Specific gravity of the source material was found to be 2.7 x $10^{11}$ with a total cost of Pakistani rupees one thousand per ton (Rs: 1000/ton).

*Figure 1: Local source material from Mattani Azakhel.*

*Figure 2: Thermal activation of source material at $M_{700}$, $M_{650}$, $M_{600}$, $M_{400}$, and $M_{200}$ (M = temperature, left to right).*

Source material was first dried at 105 °C for 1 hour in order to remove moisture contents. After complete drying source material was grinded in the range of 200 to 250 mesh size. Thermal activation of source material was carried out in muffle furnace at different temperature i.e. 200 °C, 400 °C, 600 °C, 650°C and 700 °C as shown in Figure 2. X-Ray Diffraction and Florence techniques were used to investigate that the source material are kaolinite and metakaolinite. It was found that kaolinite was converted into metakaolin perfectly at 700 °C with no single hydroxyl group.

Geopolymerization is referred to the chemical activation of metakaolin in proper ratio. Sodium hydroxide and sodium silicate is considered a perfect binder for metakaolin. The alkaline activator name water glass which have mass composition of $Na_2O$=14.7%, $SiO_2$=29.4% and water=55.9% and 98% Pure NaOH was used as binding material for source material. Ratio of Sodium silicate and sodium hydroxide was fixed at 2.5 and 18 M of sodium hydroxide.

### *2.2 SYNTHESIS OF INORGANIC CERAMIC MEMBRANE*

Geopolymerization was used to synthesize inorganic ceramic membrane. Metakaolin was produced through calcination of Kaolinite material (obtained from local reservoir) at 700 °C as shown in Figure 2. Particle size of the source material was grinded up to 9.08 µm using wet grinding technique. Geopolymeric paste was prepared by chemical activation of metakaolinite material which was done by the help of Sodium silicate and sodium hydroxide solution in fixed ratio. Membrane disk was prepared from geopolymeric paste after molding and pressing through load machine. Stainless steel of 10 mm diameter mould was filled from geopolymeric paste and press with equal distribution pressure of 46.55 MPa for 1 minute. After pressing, membrane was kept in open environment for 4 days to gain rigid shape. Thickness of 2.33 mm membrane was prepared, that have a compressive strength of 53 MPa. To achieve crystal membrane of high compressive strength synthesized membrane was subjected to manful furnace for curing and hydrothermal treatment for 15 hours at 90 °C.

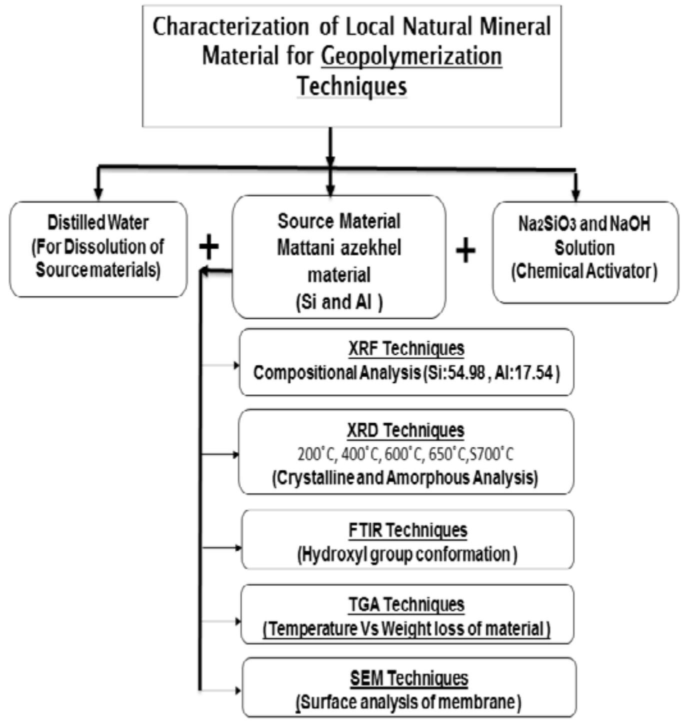


*Figure 3: Characterization of natural mineral material for geopolymerization techniques.*

## 3. RESULT AND DISCUSSION

### *3.1 MEMBRANE CHARACTERIZATION:*

Surface and cross sectional morphology of inorganic ceramic membrane was done through scanning electron microscopy (SEM) to analyze (i) The structural density of membrane before and after hydrothermal treatment, (ii) Particle size distribution and (iii) Porosity of membrane and asymmetric nature of membrane. Removal of functional group through calcination in kaolinite material was analyzed through FTIR techniques, while figure print view of metakaolin was obtained through XRD techniques. Compressive strength of synthesized inorganic ceramic membrane was done through universal testing machine.

### *3.2 SCANNING ELECTRON MICROSCOPE (SEM)*

In order to investigate the surface and cross sectional morphology of the produced inorganic ceramic membrane, scanning electron micro-graphs (SEM) were taken. SEM is the fast and nondestructive technique, which provides all the information of particle size, particle distribution, porosity and orientation of pores in surface of the membrane.

In this research work, about 200 pores of different diameters was found in the area of 10 $mm^2$ from the particle size of source material 9.43 μm. No possible defects were observed throughout SEM analysis as shown in Figure 4, of different resolutions. Average pore size was decrease and compaction occur comparatively, as on curing and hydrothermal treatment. The reason is that overlapping occurs after thermal treatment of the surface.

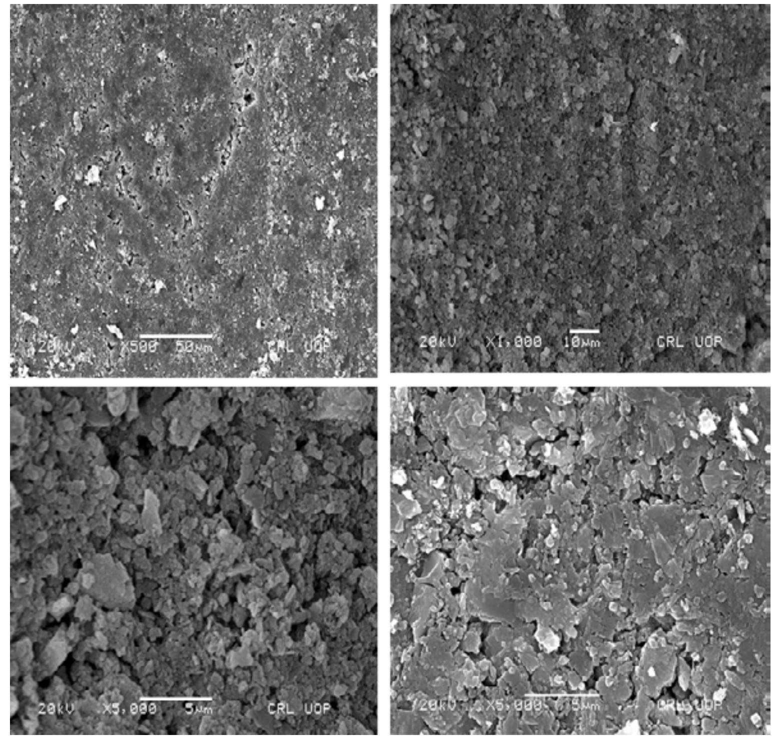

*Figure 4: SEM images of the geopolymer membranes from local mineral material.*

### *3.3 CHARACTERIZATION OF SOURCE MATERIAL*

Elemental composition of source material was investigated through x-rays Florence techniques. X-ray Florence techniques were carried out to check that the locally available mineral material would act as a source materials for geopolymerization. In Source material greater aggregate of silicon, aluminum and ferrous oxide was found as present in Kaolinite material. This compositional analysis of the material was quiet similar to the one which has been reported in the literature by Cui Xue-min [14]. Si to Al ratio affect significantly the mechanical properties of the membrane.

The percent ratio of Si to Al was found in the source material is 3.13. The local mineral material has a strong ability to act as kaolinite material in geopolymerization as shown in Table 1. P. Duxson et al, reported in his research work that compressive strength is highly dependent on compositional fraction of source material, while the optimum for high compressive strength is 3.0.

*Table 1: Chemical composition of mineral material.*

| $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $Na_2O$ | $K_2O$ | L.O. I. |
|---|---|---|---|---|---|---|---|
| 54.98 | 17.54 | 5.33 | 5.40 | 0.20 | 0.95 | 0.09 | 14.50 |

### *3.4 X- RAY DIFFRACTION (XRD)*

In addition to SEM and X-rays Florence techniques, XRD was also conducted in order to investigate XRD is one of the most reliable and advance technique to check crystalline and non-crystalline nature of materials. For structure illumination and chemical composition, XRD is considered the powerful and nondestructive analytical techniques. XRD spectrum of the mineral material from local reservoir is shown in Figure 5.

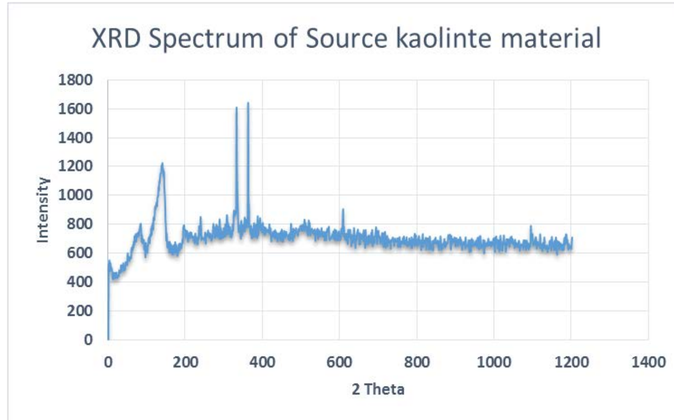


*Figure 5: XRD Spectrum of Source Kaolinite material*

During thermal activation from the range of 400 to 600 °C it was observed during XRD analysis that main constituent of source material are $SiO_2$, $Al_2O_3$ and $Fe_2O_3$ which are convert into sillimanite ($Al_2SiO_2$), corundum ($Al_2O_3$) and $Al_8Fe_2Si$ at the intensities of 1134, 1192, 898, 785 and 1216 respectively.

Further, increasing temperature from 600 °C to 700 °C, silicon dioxide was in high ratio amongst all the component in the region of 250 to 300(2 θ), and the d-value of 6.456, 5.231 and 3.192 as shown in Figure 6.

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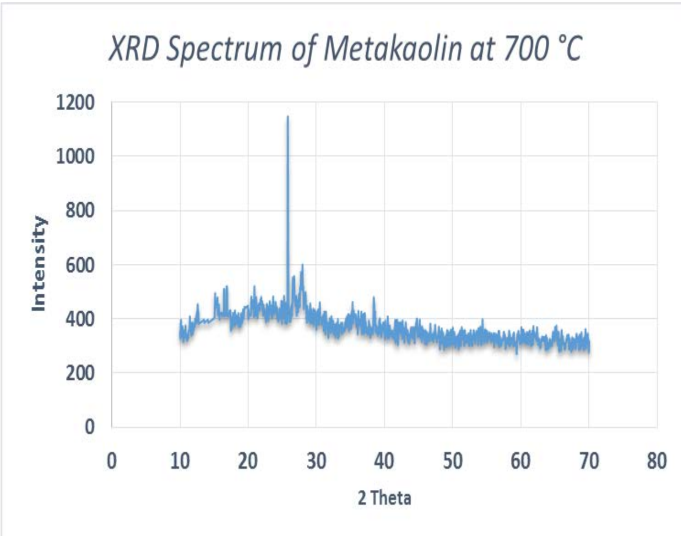


*Figure 6: XRD Spectrum of Source material at 700 °C temperature.*

### 3.5 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

The series of spectra were started from the range of 4000 $cm^{-1}$ to 380 $cm^{-1,}$ Functional group are lies in the region of 4000 $cm^{-1}$ to 1300 $cm^{-1}$, and the figure print of metakaolinite material are also lies in the same range. Linkage of hydroxyl group was pointed at the IR band of 3622.5 $cm^{-1}$. The raw sample show hydroxyl group at IR band of 3603, 3344.6, 1631.78 $cm^{-1}$. While on calcination stretching on H-O-H and Al-O-H reduced and functional group leave the source material as shown in figure 7 at different temperature.

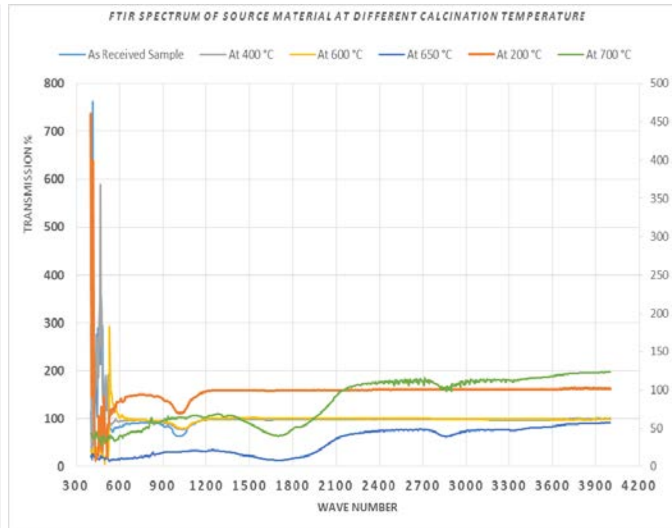


*Figure 7: FTIR Spectrum of Source material at different calcination temperature.*

### 3.6 THERMAL GRAVMATRIC ANALYSIS

Thermal gravimetric analysis was carried out to measure the thermal stability of the source material. During thermal activation at different temperatures (T °C = 200, 400, 600 and 700) was carried out for thermal stability of mineral material. Figure 8, shows clearly, that variation in weight loss of mineral material decrease with increasing calcination temperature. At 700 °C variation in weight loss on thermal treatment is negligible. Percent weight loss of source material was recorded 10.23 to 5.12 in the range of 200 to 400 °C. Small weight loss on 600 °C, while straight cure obtained at 700 °C.

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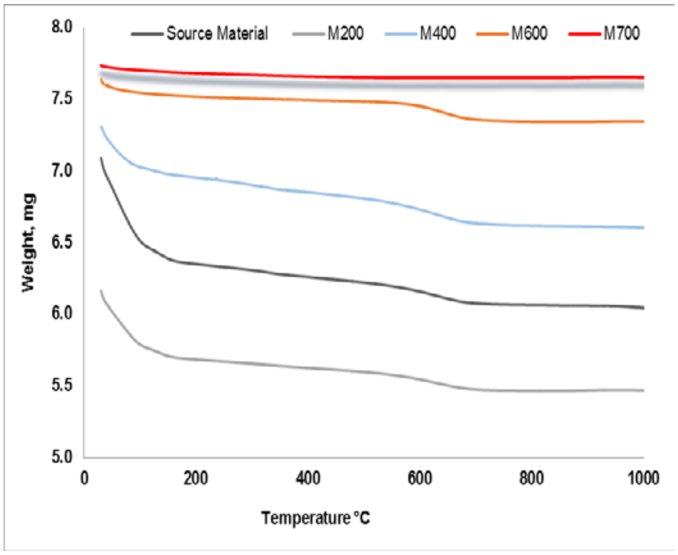


*Figure 8: TGA Spectrum of Source material at different calcination temperature.*

## 4. CONCLUSION:

From the current research work, it is concluded that locally available material from Mattani Azakhel can be used as a source material in geopolymerization techniques. It was found from XRF techniques that local mineral material is highly in rich with silicon and aluminum. From XRD pattern it was confirmed that this mineral material provide a pattern as kaolin. Mostly kaolin material calcinated at above 750 °C, while locally source material calcinated at low calcination temperature of 700 °C. Synthesis of ceramic membrane with free sintering temperature is only possible through geopolymerization techniques. High compressive strength of 62 MPa inorganic ceramic membrane is synthesized using the same mineral material. Synthesized membrane was defect free with pores size of 0.8 to 0.9 µm. Inorganic ceramic membrane synthesized through geopolymerization stand promise for waste water filtration to remove bacterial and other microbial animals.

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