utilization of kaolin to produce zeolite

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UTILIZATION OF NAGARPARKER KAOLIN FOR THE SYNTHESIS OF ZEOLITE-ZSM, A VALUE ADDED PRODUCT

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Abstract

Nagarparker arid zone of Pakistan. It has large deposits of Kaolin (China Clay) approximately 4.3 million tons. Crystal zeolite ZSM produced and characterized. Material ground, calcined dehydrated using ball mill, autoclave and furnace respectively due to dehydration kaolinite converted into metakaolin with a weight loss of about 3.9% and 8 M solution of Sodium hydroxide added with the ratio of 1:5 at 100 °C with vigorous stirring for 1 h. Fusion carried out at 100 °C for 1 h, sample washed 3 times to make its pH normal. Characteristic Si-O-Al, OH, Al-OH and Si-OH bands were confirmed in Fourier Transform Infrared Spectroscopy studies. Scanning Electron microscopy showed clear morphology of zeolite zsm. Xray Diffraction showed 2Θ peaks and revealed orthogonal shaped crystal structure. The results show that the Nagarparker kaoling is suitable for the synthesis of Zeolite ZSM as value added Product.

Keywords: Nagarparker Kaolin, Synthesis, Zeolite-ZSM, Utilization

1. INTRODUCTION

Nagarparker is the region containing reserves of approximately 3.67 Million Tons (Kella 1983). Muslim et at reported geological map of Nagar Parkar area in 1997 (Muslim, Akhtar et al. 1997). Kaolin deposits as shown in Figure 1. vary from 1.50 to 10.0 m in thickness and average overstretch thickness is 2.10 m (Ismail, Husain et al. 2019). Clay minerals like kaolinite, sepiolite, mica, palygorskite, smectite and vermiculite groups are naturally occurring minerals. Out of these kaolinite ($Al_2Si_2O_5(OH)_4$) is one of the most important material to be used for the production of zeolites (Konta 1995, Murry 2000, Franco, Pérez-Maqueda et al. 2004). Zeolites are found naturally (Abdollahi, Towfighi et al. 2020, Moazeni, Parastar et al. 2020) and can be made synthetically(Goodarzi, Herrero et al. 2020, Vinaches and Pergher 2020, Wei, Wang et al. 2020).

Kaolin is obtained from silicate rocks as a weathering product of silicate its color varies from whitish powder to earthy having some degree of plasticity. It is an industrial mineral used as base material for ceramics and production of refractories, sometimes used as material for industrial filling of paints, rubber, plastic, dyes and paper (Konta 1995, McClendon 1999, Franco, Pérez-Maqueda et al. 2004). Additionally, kaolin can be utilized for waste management (Osmanlioglu 2002, Bhattacharyya and Gupta 2008) and in the preparation of geopolymers and geopolymer-based composites (Xu and Van Deventer 2002, Wang, Li et al. 2005) zeolites (Meftah, Oueslati et al. 2009, Rios, Williams et al. 2009) and interpolates (Pinnavaia and Beall 2000, Letaief, Elbokl et al. 2006).

Kaolin is a natural mineral from the clay family and can contain a number of impurities, like zircon, feldspar, quartz, tourmaline etc., which are obtained from the parent rock. Initially properties and structure of kaolinite was studied by Brindley and Robinson (Brindley and Robinson 1946). They determined lattice parameters by analysis of reflections using patterns obtained from X-ray diffraction. Karmous (Karmous 2011) showed lattice energy -827.4eV in kaolinite by applying technique of computational energy minimization. The total lattice energy in kaolinite is equal to standard. While the primitive cell volume is 321.30 Å. Kaolinite elastic constants were calculated by using first principle calculations by Militzer*et al*., (Militzer, Wenk et al. 2011). Also discussed possible application areas of kaolinite. With Rietveld refinement, Young and Hewat (Young and Hewat 1988).

Figure 1. Map of Pakistan Showing Nagarparkar Kaolin deposits.

Though a few researchers, like Edomwonyi-Out *et al*. (Atta, Ajayi et al. 2007, Edomwonyi-Otu, Aderemi et al. 2013, Bawa, Ahmed et al. 2016) have explored Kankara kaolinite of Nigeria by checking its influence of thermal treatment. Possible application areas of kaolinite deposits and its in-depth properties are still lacking. Worldwide kaolin production is $38*10^6$ Metric Ton. Literature suggest that kaolin is best material to produce various types of zeolite like Mia synthesized zeolite-A (Maia, Angélica et al. 2011), and activated waste kaolin (Maia, Angélica et al. 2014).

Ethopian kaolin was used to produce zeolite-A (Ayele, Pérez-Pariente et al. 2015) used it as additive for detergent (Ayele, Pérez-Pariente et al. 2016) and fro the removal of Chromium (Cr) (iii) from tannery waste (Ayele, Pérez et al. 2018). Irani kaolin used by irani to produce zeolite-zsm5 (Khatamian and Irani 2009). Jordion kaolin was used to produce zeolite A (Gougazeh and Buhl 2014). Tunisian kaolin was was studied by (Felhi, Tlili et al. 2008) and used to produce zolite LTA (Tounsi, Mseddi et al. 2009) and NaX zeolite (Ghrib, Frini-Srasra et al. 2016). Literature suggests use of kaolin to synthesize zeolite (Ugal, Hassan et al. 2010, Amber, Folayan et al. 2013). Ugal *et al*., (Ugal, Hassan et al. 2010) synthesized zeolite 4A from Iraqi kaolin. Ion exchange technique was used to insert sodium to the structure and water adsorption purposes were identified by conducting tests ont the zeolite. Zhao *et al*., (Zhao, Zhang et al. 2010) also used halloysite mineral to produce well-ordered NaA zeolite (Kovo, Hernandez et al. 2009, Mgbemere, Lawal et al. 2018). Literature survey don't suggest any of zeolite zsm production using Ngarparkar kaolin reserves found in Pakistan.
The aim of this research is thus, to identify the characteristics (composition, structural order etc.) of kaolin samples obtained from Nagarparker Pakistan. It also aims to produce zeolite-zsm form the samples of kaolin and to compare the results of obtained zeolite-zsm with the zeolite-zsm samples in the literature.

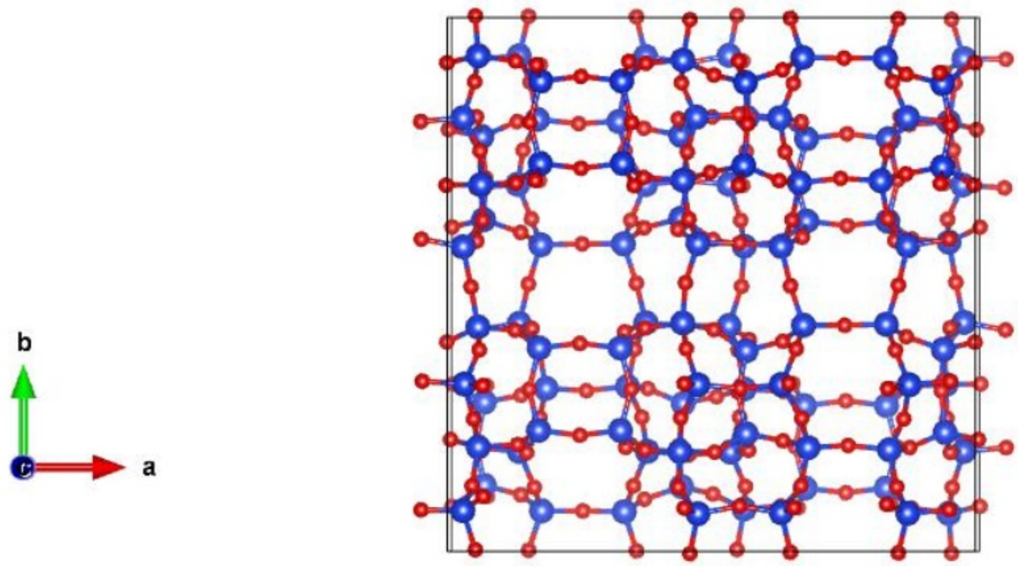

Figure 2. Single component Crystal structure of Zeolite ZSM obtained from International Zeolite Association.

2. METHODS AND MATERIALS

2.1. Material collection

Samples of kaolin were collected from Nagarparker area of Pakistan having longitudes and latitudes Sindh (24º 15' to 24º 30'N, 70º 37' to 71º 07' E. Major Oxides of Nagarparkar kaolin is mentioned in Table1. Physical properties are such as density, specific gravity, plastic limit, Liquid limit, Plastic index and shrinkage index are given in Table 2.

2.2. Calcination

Calcination of Kaolin clay was done for 4 h at 700 °C. Due to this clay was activated and metakaolinite was formed with the loss of 3% by weight %. Alkali-activated paste was made of 8 Molarity by adding NaOH in to water. Thus, a more active amorphous metakaolin with a small amount of quartz was obtained as a product. The alkali-activated paste samples were synthesized using 4 ml of a 5-10 M solution of NaOH and 5 g of metakaolin. Mechanically the solution was mixed with solids at room temperatures for several minutes. The fresh pasta was poured into a silicone mold and activated at 100 ° C. for 4 hours.

2.3. Gel formation

The autoclave was filled with various concentrations of sodium hydroxide, distilled water and metakaolin were added to obtain a solution. A gel-like glossy solution was obtained. Agitation and aging Agitation at 90 °C for 60 minutes

2.4. Crystallization and post treatment

The gel was crystallized at 100 °C. for 24 hours. After crystallization, filtration and washing were carried out with water demonized from zeolite crystals obtained until the pH was lower than 10.

Figure 3. Zeolite Synthesis from Nagarparkar kaolin

2.5. Characterization

the starting material and synthesized product was characterized by FTIR, SEM, XRF and XRD techniques, as follows. X-ray powder diffraction (XRD) analyses were carried out with a P Analytical X'Pert Pro MPD (PW3040/60) diffractometer applying linear detector by high-speed solid-state linear (X'Celerator). using Cu-Ka radiation (l = 1.5406 A°) on powdered samples in y/y scanning mode randomly Ni Kb filter. X-ray powder patterns were collected. The scan range was 5º to 75º 2y, with the following instrumental conditions: 1/4º anti-scatter slit, 1/8º divergent slit, Fourier transform infrared spectrometry (FTIR) employed a Perkin Elmer 1760 X FTIR spectro-meter in the 4000-400 cm-1 range with samples prepared as KBr discs.

Scanning electron microscopy (SEM) analyses were carried out on a Zeiss LEO 1430 microscope. The samples were previously sputtered with gold using Emitech K 550 equipment 0.02º 2y step size and 20 s per step.

Table 1. Composition of major oxides of raw and washed kaolin of Nagarparker.

Oxides	SiO_2	TiO_2	Al_2O_3	FeO_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	LOI
Raw Kaolin of Nagarparkar	52.9	1.3	24.7	0.7	0	0.2	4.8	1.4	0.3	0	13.7
washed kaolin of Nagarparkar	45.1	0.7	35.1	0.7	0	0.6	1.8	1.2	0.2	0	14.8

Table 2. Physical properties of Nagarparker kaolin

Locality name	Density g/cm3	Sp. Gravity	Plastic Limit%	Liquid Limit %	Plastic Index %	Shrinkage Limit %
Nagarparkar kaolin	2.53	2.54	24.9	39.5	14.61	1.67

3. RESULTS AND DISCUSSIONS

3.1. XRD pattern

Phase compositions were analyzed by Xray Powder Diffraction (XRD) X'Perts system (CuKα radiation). Range of 2θ degree angle of 5-90° with a step of 0.007 for 2 hr. X'Pert HighScore Plus application was used to identify phases with the use of an and the International Centre for Diffraction Data. The bands intensity ratio of 550 and 450 cm-1 was utilized to estimate the crystallinity of zeolite. Considering value of 0.70 for well crystalline ZSM, the observed value of 0.68 for the products as the XRD graph is presented in Figure 5 which clearly suggests that the framework of the zeolite.

3.2. FTIR Analysis

Fourier Transform and Infrared Spectroscopy was done of raw kaolin obtained from Nagarparkar named as sample hk1 and the product synthesized called zeolite zsm is shown in Figure 4 and Figure 5 respectively. 1400-400 cm-1 are wavenumber regions of solid-state reaction product. The course of the spectra was subsequently confronted with structure and the character of bonds in these zeolites

shift of the band at around 1435 cm-1 toward higher wavenumbers can be observed with the products of the solid-state reaction, indicating that the reaction has slightly influenced the bonding strength of the zeolite framework.

Figure 4 describes clearly that (Al, Si) O bonds were estimated by tetrahedrons stretching vibrations at wavenumbers 400-1100 cm-1 for the kaolin samples obtained from locality of Nagarparker. OH groups of water molecules were also observed through stretching vibrations at wavenumbers 1590-1670 and 3400-3700 cm-1. Wavenumbers 300-500 and 1100-1250 cm-1 having external vibrations shows (Al, Si) O. tetrahedrons.

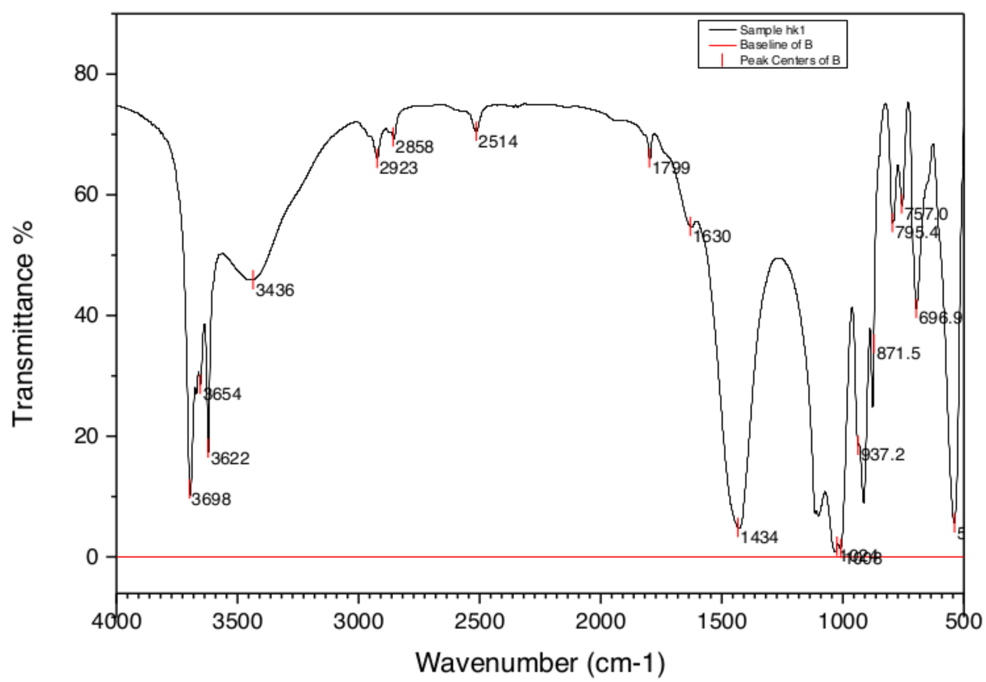

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Figure 4. FTIR Spectrum of Nagarparkar kaolin

In Figure 5 the tetrahedrons vibrations were observed (wavenumbers 300-500 and 1100-1250 cm-1) Figures 5 containing three parts (a), (b), and (c) shows the FTIR spectra of the products obtained after 1h. Bands at around 3600 cm-1 and 1600 cm-1, attributed to zeolitic water, were observed in all the products. Also, a band at around 1000 cm-1 was observed, characteristic of the Si-O-Al bonds in TO_4 tetrahedra, which confirmed the presence of zeolitic material. Another band was observed at around 531 cm-1 for all the products obtained. Also, a band at around 1000 cm-1 was observed, characteristic of the Si-O-Al bonds in TO_4 tetrahedra (Mozgawa, Sitarz et al. 1999), which confirmed the presence of zeolitic material. Another band was observed at around 531 cm-1 for all the products obtained.

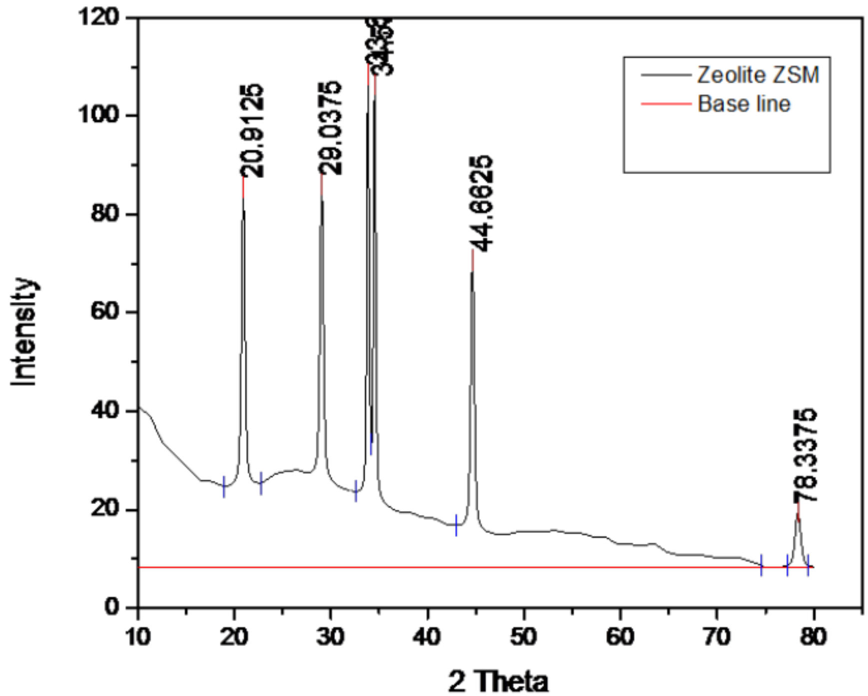

Figure 5. XRD Pattern of Zeolite ZSM produced from Nagarparker kaolin.

3.3 SEM Analysis

Scanning Electron Microscopy analysis were taken at three magnifications (a) at x2500, (b) at x5000 and (c) at x10000. Morphology clearly indicate presence of particles with pores. Direct imaging technique was applied on the surfaces of particles of zeolite, types of pores existing on are revealed by SEM technique. Distribution of atoms on zeolite can by visualized on their surfaces by conjunction analysis by cross-sectional observations as described in Figure7.

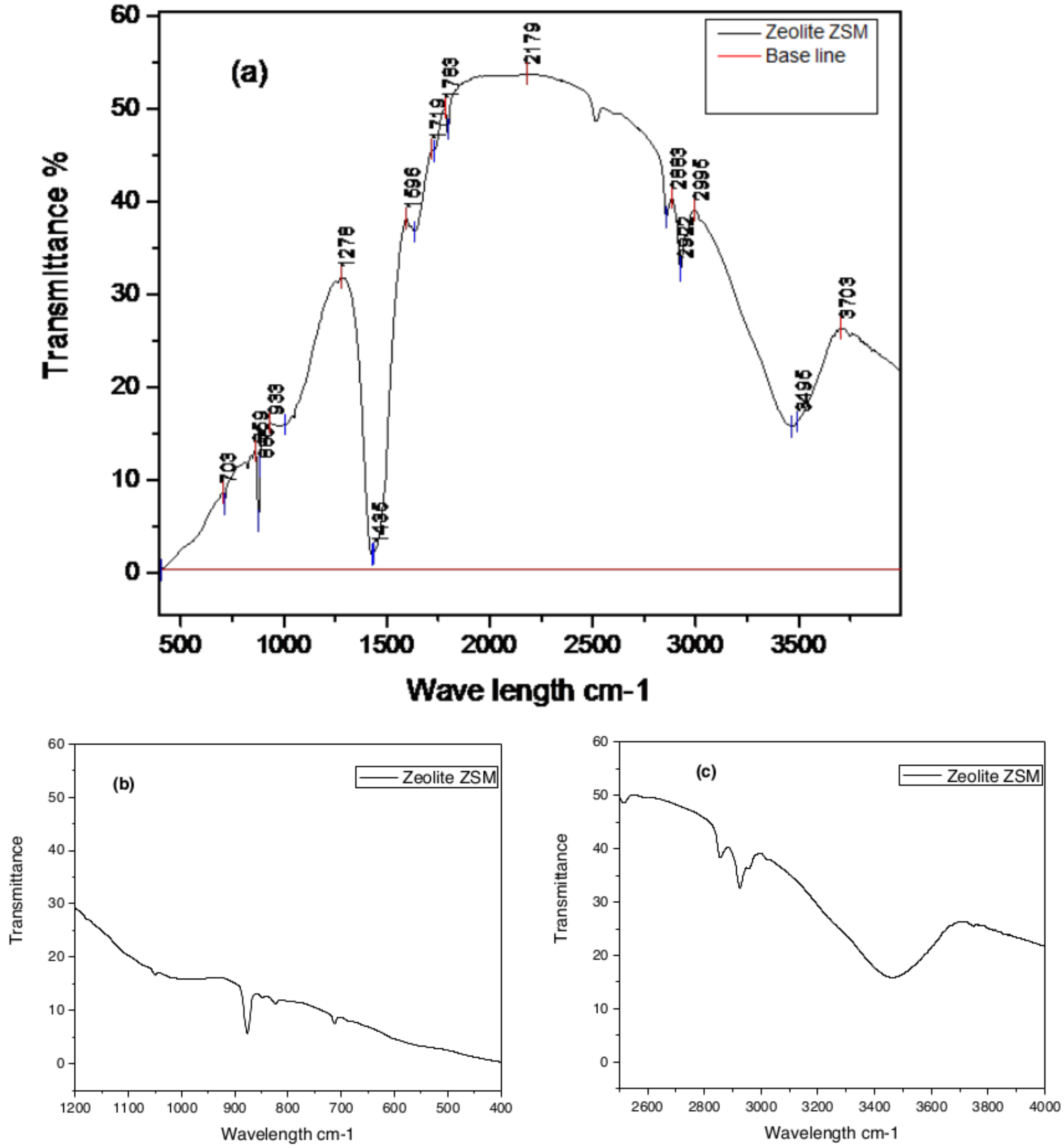

Figure 6. FTIR Pattern of Zeolite ZSM prepared from Nagarparker Kaolin. (a) Zeolite ZSM prepared from Nagarparker kaolin. FTIR range of wavelength is from 1200 cm-1 to 400 cm-1. (c) Zeolite ZSM prepared form Nagarparker kaolin. FTIR range of wavelength 2500 cm-1 to 4000 cm-1.

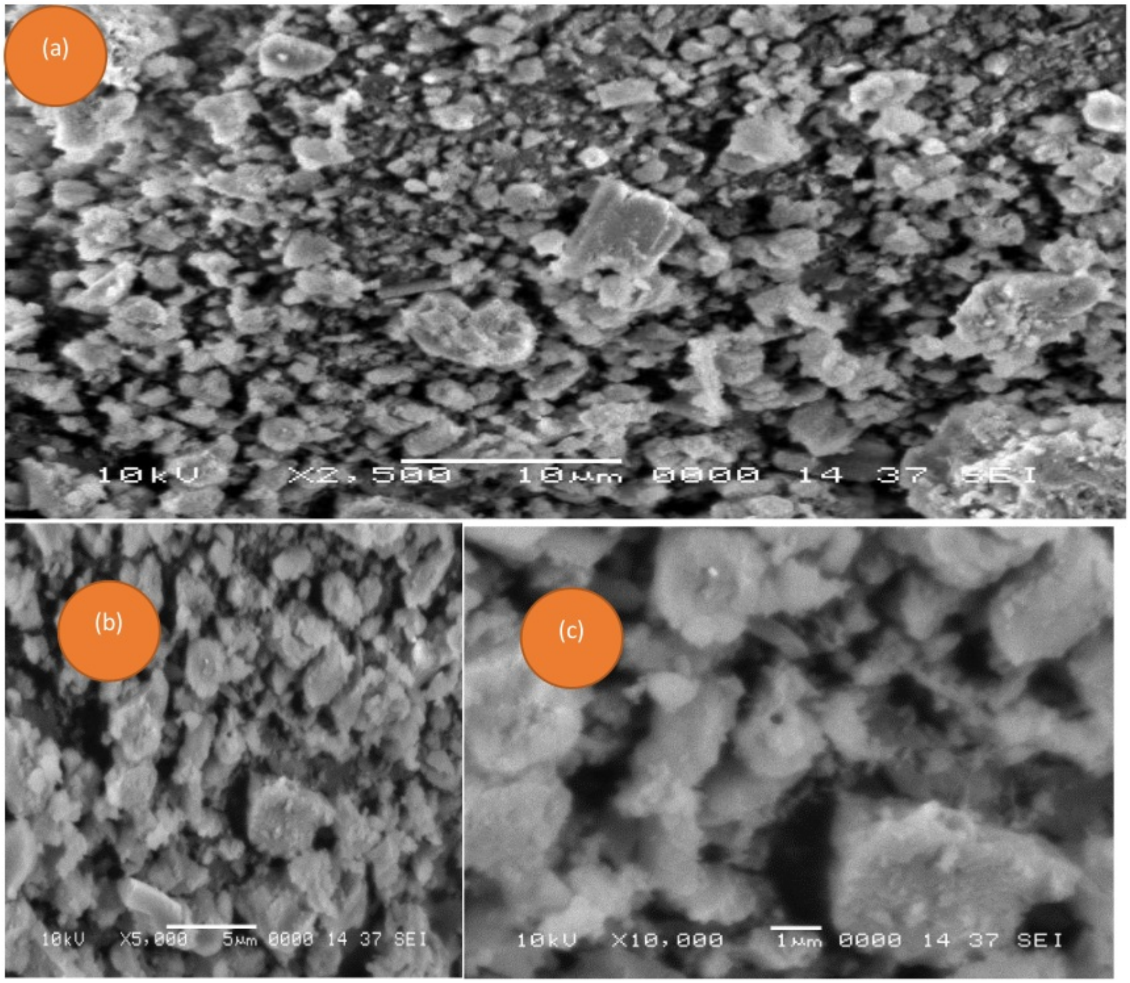

Figure 7. Morphology of Zeolite ZSM produced from Nagarparker kaolin (a) Resonance focus of X2500 (b) Resonance focus of x5000 and (c) Resonance of x10000

4. CONCLUSION

The Nagarparker kaolin of Pakistan consists mainly of kaolinite. Anataze, Quartz, and muscovite are auxiliary minerals in the kaolin. They are removed during processing (magnetic separation, centrifuga-tion, etc.) and dumped into kaolin waste basins. Zeolite-ZSM was synthesized using the kaolin waste as starting materials.

Kaolin of Nagarparker can be easily used as basic material for synthesis of zeolite-ZSM. Importantly, there is a significant environmental advantage to use unexplored reserve of kaolin as raw materials. The value of reserve can be enhanced by such a scientific approach. Different industrial purposes can be achieved by producing zeolites. Definitely it would aid realistic economic and social development of the region.

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