

Effect of modification of Zeolite A using Poly Vinyl Alcohol (PVA)

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Structural modification of raw zeolite A was carried out at a temperature of 80°C using low and high Poly Vinyl Alcohol (PVA). The product was characterized by X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-ray Spectroscopy (EDS), High Resolution Transmission Electron Microscopy (HRTEM) and Image Analyzer (IA). XRD shows PVA anchored to the faces [6 0 0], [6 2 2], [6 4 2], [6 4 4] resulting in a decrease in the crystallite size of the modified zeolite. But 600°C calcination cannot remove the anchored PVA from the zeolite faces, so calcination does not change the crystallinity. FTIR shows the noise level of the bands in the region 400-420 cm⁻¹ decreased in modified zeolite A indicating the raw zeolite surface is smoothened by the application of PVA. From FESEM it is found that the shape of the modified particles changed to slightly spherical with the reduction in size. EDS confirms the oxygen percentage is increasing whereas the Na, Al, and Si percentages are decreasing after modification. HRTEM shows the modification is prominent for zeolite modified with a high PVA and the particle size is reduced to 1.5µm. The particle size analysis shows that after modification with a low and high PVA the maximum particles have the size within the range of 2-2.5 µm (29 % size reduction as compared to raw zeolite A) and 1.5-2 µm (43 % size reduction as compared to raw zeolite A) respectively and there was no significant change in particle size after calcination.

Keywords: Zeolite A, Poly Vinyl Alcohol (PVA), Ultrasonication, Centrifugation and Calcination.

INTRODUCTION

Zeolites are a crystalline hydrated framework of aluminosilicate having group I and II elements, in particular, sodium, potassium, calcium, magnesium, strontium and barium [1, 2]. More than 150 synthetic and 40 naturally occurring zeolites are known [3]. They can be represented by the empirical formula $M_{2/n}O \cdot Al_2O_3 \cdot xSiO_2 \cdot yH_2O$. In this oxide formula, x is generally equal to or greater than 2, since tetrahedral AlO_4 joins only tetrahedral SiO_4 and n is the valency of the cation. Natural zeolites were used initially for different applications, but more recently modified and synthetic forms have been made on an industrial scale giving rise to tailor made zeolites that are highly replicable. Zeolites are highly crystalline due to the well defined structure and enclose the aluminium silicate framework cavities occupied by large ions and water molecules, with the cavities opening ranging from 0.8-1.0 nm in diameter which are the order of the molecular dimensions. The size and shape of these pores determine which molecules enter the cavities and which are excluded. These are called molecular sieves [4]. Cations within the cavities are easily replaced with a large number of different valency cations which exert large electrostatic forces across the small dimension of the cavity, while the introduced cations have separate activities

of their own; this facilitates dual function catalysis involving acidity along with other activities [4].

The chemical formula of zeolite A is $Na_{12}[AlO_2 \cdot SiO_2]_{12} \cdot 27H_2O$. According to the data base for zeolite structure [5], zeolites of type A are classified into three dimensional grades, 3A, 4A and 5A, all of which possess the same general formula but with a different cation type. Zeolites commercially produced from sodium aluminate and silicate by hydro gel processes [6]. Faujasite zeolites are obtained from KanKara Kaolin clay [7] and zeolite NaX was synthesized from Kerala Kaolin [8]. Because of the presence of a large volume of micro pores and the high thermal stability of the zeolite, this material is used for purification of waste water, and soil remediation [9-10]. Synthetic zeolites are widely used as industrial adsorbents for various gases and vapours [8] and as catalysts in the petroleum industry [11]. Zeolites have a high tendency to adsorb water and other polar compounds like NH_3 , CO_2 , H_2S and SO_2 and have a good capacity at very low temperatures compared with other adsorbents. Pressure swing adsorption (PSA) is one of the techniques which can be applied for the removal of CO_2 from gas streams. Zeolite has shown promising results in the separation of CO_2 from gas mixtures and can potentially be used in the PSA process [12-14].

Perfect defect free zeolite crystalline structures are not readily available or easy to prepare. Therefore most of the zeolite material has defects

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and spaces between the crystals which are larger than the pore sizes in the crystalline structures. To control the pore size different methods have been adopted for modification of zeolite [15-21]. A lot of work has already been done for chemical modification to prepare the composite membranes for gas separation. No extensive work has been done for physical modification of zeolite A. The authors have reported the effect of modification of zeolite A using Carboxy Methyl Cellulose (CMC) [22].

The present study focuses on the physical modification of zeolite A to reduce particle size, as well as to achieve a uniform distribution. There are different types of polymer hydro gels having temperature dependent gelation behavior, i.e., they convert to gel at an elevated temperature and turn back to solution at room temperature. Poly Vinyl Alcohol (PVA) is a polymer that is cheap, economical, water soluble, eco-friendly and adheres onto zeolite A. Hence a hydro gel surfactant PVA was used for the modification of zeolite A.

EXPERIMENTAL METHOD

Materials

Raw zeolite A purchased from NALCO, India was used as the starting material for the modification experiments. The chemical composition was determined by Atomic Absorption Spectroscopy (AAS) using Perkin Elmer AAnalyst 200/400, as shown in Table 1. Ignition loss and pH (1% in water) were found to be 21.84% and 10.3, respectively.

Table 1. Composition of Zeolite A

Molar composition (Based on chemical analysis)	Average Chemical Composition (%)
1.0 ± 0.2 Na ₂ O	Na ₂ O 16.5-17.5
1.0 Al ₂ O ₃	Al ₂ O ₃ 27.5-28.5
1.85 + 0.5 SiO ₂	SiO ₂ 32.5-33.5
6.0 (Max.) H ₂ O	

PVA was purchased from the Central Drug House (CDH), India with the specification of technical purity (99.5 %).

Modification of zeolite

Zeolite A was modified by using low and high PVA as per the concentration. To modify zeolite using low PVA about 0.17 g of PVA was taken in a beaker, 150 mL of de-ionized water was added and ultrasonic dispersion was carried out in an ultrasonic bath attached to an ultrasonic generator (TPC-15H, Telesonic AG, Switzerland) having a peak power of 150 watt for a period of 5 minutes

under a frequency of 30 kHz so as to make a homogeneous solution. Then 9.65 g of zeolite A was added to the solution. Ultrasonic dispersion was carried out for 3 h at 80°C. Finally the zeolite was recovered from the mother liquor by repeated cycles of centrifugation, decanting and ultrasonic redispersion in pure water until PVA was completely washed away (no bubbles observed). Modified zeolite was dried at 100°C for 3 h and calcined for 4 h at 600°C. The same procedure was adopted using 6.5 g of PVA with 5 g of raw zeolite for modification of zeolite using high PVA.

Characterization

The crystalline structure of the modified zeolite A was determined by X-ray diffraction using a PANalytical XPERT-PRO diffractometer with Cu-K α radiation ($\lambda=1.5406\text{\AA}$). Diffraction measurements were performed over the 2θ range from 5-80°.

The functional groups present after modification of zeolite A were determined by Fourier transform infrared spectroscopy (FTIR) using a Perkin Elmer SPECTRUM-GX FTIR spectrometer in the 4000-400 cm⁻¹ region using pellets of 0.5 mg powdered samples mixed with 250 mg of KBr.

The microstructure and the morphology of size reduction of the modified zeolite A were examined using field emission scanning electron microscopy (FESEM model ZEISS EM910).

The compositions of the modified zeolite A were examined by energy dispersive X-ray spectroscopy (EDAS model ZEISS EM910).

The particle size of the modified zeolite A was determined using high resolution transmission electron microscopy (HRTEM model ZEISS EM910) operated at 100 Kv, with a 0.4 nm point-to-point resolution side entry goniometer attached to a CCD Mega Vision III image processor.

The particle size distributions were examined by using an image analyzer (IA model SAMSUNG SDC-313B) to determine the amount of reduction of the particles and distributions of the particles.

RESULTS AND DISCUSSION

The powder X-ray diffraction patterns of raw, water treated, low and high PVA modified zeolite A are shown in Fig. 1(a),(b),(c) and (d) respectively.

The patterns are plots of the X-ray intensity scattered from the sample versus the scattering angle (Bragg angle, 2θ). The positions and intensities of the peaks in the diffraction pattern are a fingerprint of the crystalline components present in the sample. In the samples Na₂O, Al₂O₃ and SiO₂ planes are present in the orthorhombic,

rhombohedral and hexagonal unit cells, respectively. The faces [6 0 0], [6 2 2], [6 4 2], [6 4 4] are with higher intensities than [2 0 0], [2 2 0], [2 2 2], [4 2 0]. When treated with PVA, it anchored to faces [6 0 0], [6 2 2], [6 4 2], [6 4 4]. This is evident from the lowering of the peak intensities. The peaks in the XRD pattern of zeolite A treated with PVA are slightly broadened, as compared to those of raw zeolite A and zeolite A treated with water. This points to a decrease in the crystallite size of the modified zeolite A [15].

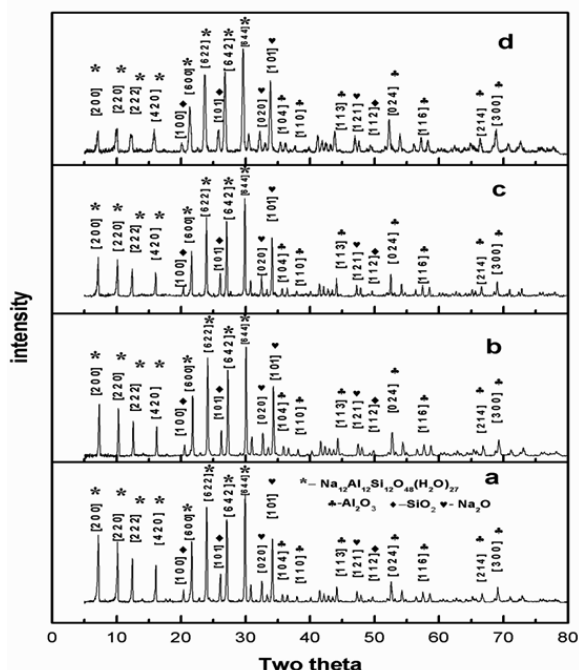


Fig.1. X-ray diffraction patterns of (a) raw zeolite A, (b) zeolite A treated with water, (c) zeolite A modified with low PVA and (d) zeolite A modified with high PVA

During modification, the temperature does not exceed 80°C. It is found that during sonication, the local heat caused by inter-particle collisions (for ~10

µm particles) could reach 2600-3400°C [23]. Thus, it is possible that the modification of the supplied zeolite A could take place at a lower macroscopic temperature because of the extremely high local temperatures generated during sonication. It is observed that sub-micron particles cannot be separated by stirring. Sonication is one of the most effective methods for dispersing the particles; whereas due to the cavitation effect the generated micro jet and shock-wave impact on the surface together with the interacting collisions result in particle size reduction; however a stabilization technique like centrifugation must be used to prevent high agglomeration once sonication is stopped. PVA is used as a surfactant, which might cover the zeolite particles to keep them in suspension and to avoid agglomeration. It was also seen that the particle size reduction strongly depends upon the frequency, amount of the sample, power supplied with the processor and time of treatment. Nevertheless the prolonged ultrasonic treatment increases the structural dis-order of the particle and temperature of the solution [24]. The higher temperature might de-mature the PVA structure and an interaction is prompted at elevated temperatures. That is why we picked up 5 minutes for sonication and 80°C as a reaction temperature well below the boiling point of the solution. Calcinations do not change the crystallinity and at 600°C calcinations cannot remove the anchored PVA from the zeolite faces, which is evident from the low-intensity peaks [1 0 1], [6 4 4], [6 2 2], [6 4 2], [1 0 1].

The FTIR Spectra of raw, water treated, low and high PVA modified zeolite A are shown in Fig. 2(a), 2(b), 2(c) and 2(d), respectively. The absorption peaks are discussed individually.

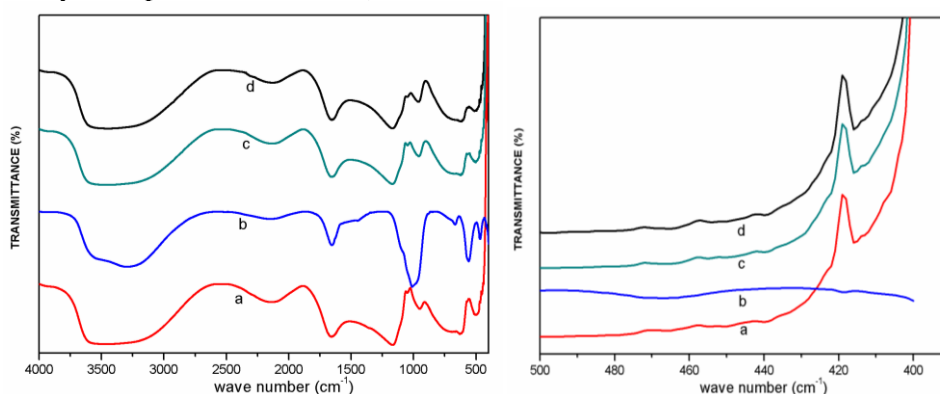


Fig.2. FTIR spectra of (a) raw zeolite A, (b) zeolite A treated with water, (c) zeolite A modified with low PVA and (d) zeolite A modified with high PVA.

A characteristic strong and broad band at 3400 cm^{-1} is seen due to O-H stretching vibrations [25]. The band at 2100 cm^{-1} is due to Si-H stretching vibration [26-27]. The deformation band at 1640 cm^{-1} confirming the presence of bound water [25], pre-dominant in Fig. 2 (b), is related to the (H-O-H) bending vibration of water molecules adsorbed on zeolite. The band at 1150 cm^{-1} appears because of Si-O-Si asymmetric stretching [28], which is insignificant in Fig. 2(b) due to the presence of excess water molecules in the pores of zeolite A treated with water. The band appearing at 1034 cm^{-1} [29] related to T-O-T (T=Si and/or Al) stretching is more intense in zeolite A treated with water as shown in Fig. 2 (b) because of the excess of water molecules. The asymmetric Al-O stretch of Al_2O_3 is located at 950 cm^{-1} [30]. The bands at 557 cm^{-1} and 620 cm^{-1} (in the region of $500 - 650\text{ cm}^{-1}$) are related to the presence of double rings (D4R and D6R) within the framework structure of these zeolites [30]. The band at 557 cm^{-1} also could represent the beginning of the crystallization of a zeolite with double rings [31]. The bands at 420 cm^{-1} and 490 cm^{-1} (in the region of $420-500\text{ cm}^{-1}$) are related to internal tetrahedral vibrations of Si-O and Al-O in SiO_2 and Al_2O_3 [30]. The two most

intense bands of the zeolite usually occur at $860-1230\text{ cm}^{-1}$ and $420-500\text{ cm}^{-1}$, as shown in Fig.2. The first is assigned to an asymmetric stretching mode and the second one to a bending mode of a T-O bond. All these bands are more or less dependent on the crystal structure. The mid regions of the spectra contain the fundamental framework vibration of Si (Al) O_4 groupings [32]. The bands in the region $400-420\text{ cm}^{-1}$ are related to the pore opening or motion of the tetrahedral rings, which form the pore openings of the zeolite [2]. This is shown in the case of raw zeolite A and zeolite treated with PVA but in the case of water-treated zeolite the bands are missing, which is clearly evident from the spectra. The noise level of the bands in the region $400-420\text{ cm}^{-1}$ decreased in the case of zeolite A modified with PVA which indicates that the rough zeolite surface is smoothed by the application of PVA.

The FESEM studies of raw, water treated, low and high PVA modified zeolite A are shown in Fig. 3(a),3(b),3(c)and3(d), respectively. The particle size of the raw zeolite A is in the range of $2.5-3.5\text{ }\mu\text{m}$ with high agglomeration, which remains unchanged in case of zeolite A treated with water.

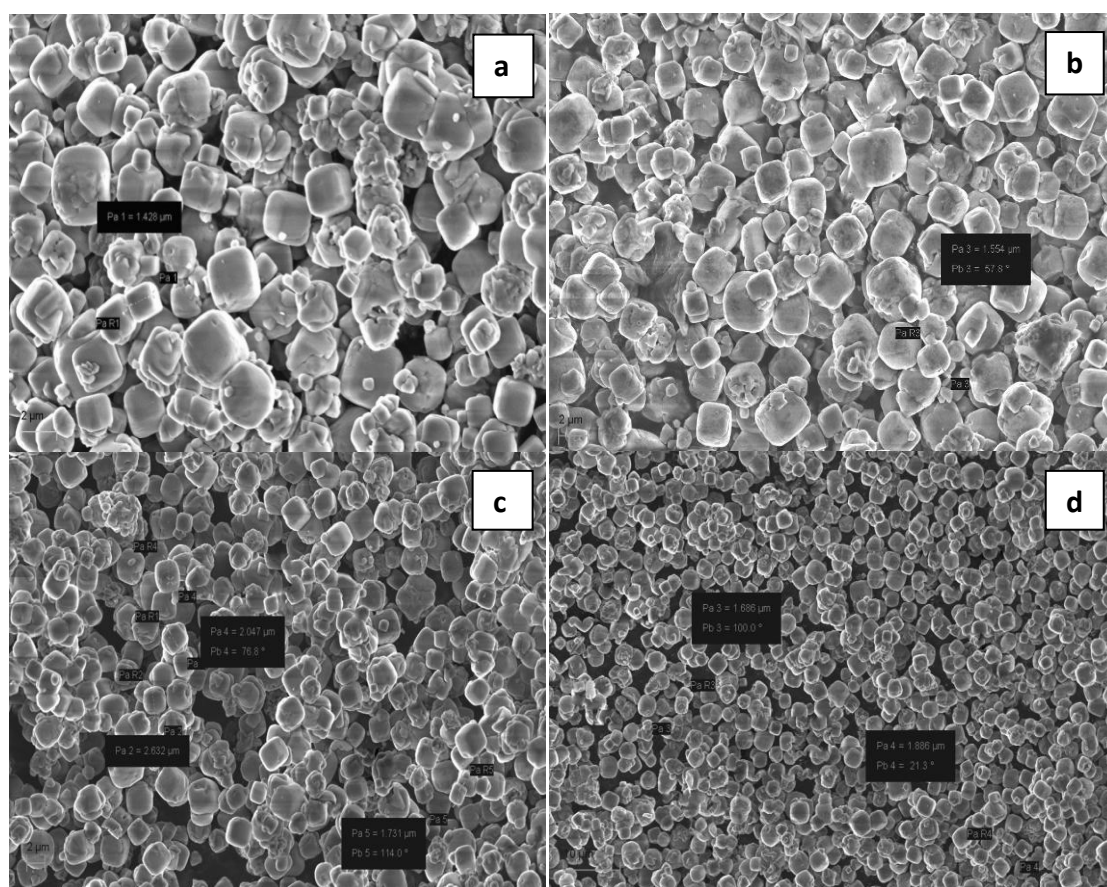


Fig.3. FESEM micrographs of (a) raw zeolite A, (b) zeolite A treated with water, (c) zeolite A modified with low PVA and (d) zeolite A modified with high PVA.

After modification with low and high PVA the particle size was found to decrease to a range of 2-2.5 μm and 1.5-2 μm , respectively with better dispersion. The particle size decrease more in case of zeolite A modified with a high PVA possibly due to exploitation of zeolite with a high PVA loading.

The shape of the modified particles changed to a slightly spherical one, as observed in Fig. 3 (d). This may be as a result of calcination.

The EDAS studies of raw, water treated, low and high PVA modified zeolite A are shown in Fig. 4 (a), 4 (b), 4 (c) and 4 (d), respectively.

The EDAS is done to determine any change of composition both raw and modified zeolite. It is seen from Table 2 that the composition, weight and atomic percentage are changing slightly. The oxygen percentage is increasing whereas the Na, Al, and Si percentages are decreasing after

modification. This may be due to the particle size reduction after modification. Further, it should be noted that in both raw zeolite A and zeolite A treated with water, the distribution of the particles is not uniform, whereas in the modified one, the particle distribution is uniform and with very few agglomerations.

The HRTEM micrograph studies of raw, water treated, low and high PVA modified zeolite A are shown in Fig. 5 (a), 5 (b), 5 (c) and 5 (d), respectively. It is seen that the particle size is in the range of 2.5-3.5 μm for zeolite A (as supplied) and remains unchanged in case of zeolite A treated with water. After modification with low and high PVA, the particle size is found to be 2-2.5 μm and 1.5-2 μm , respectively, which confirms the reduction of the size and change in shape of the zeolite.

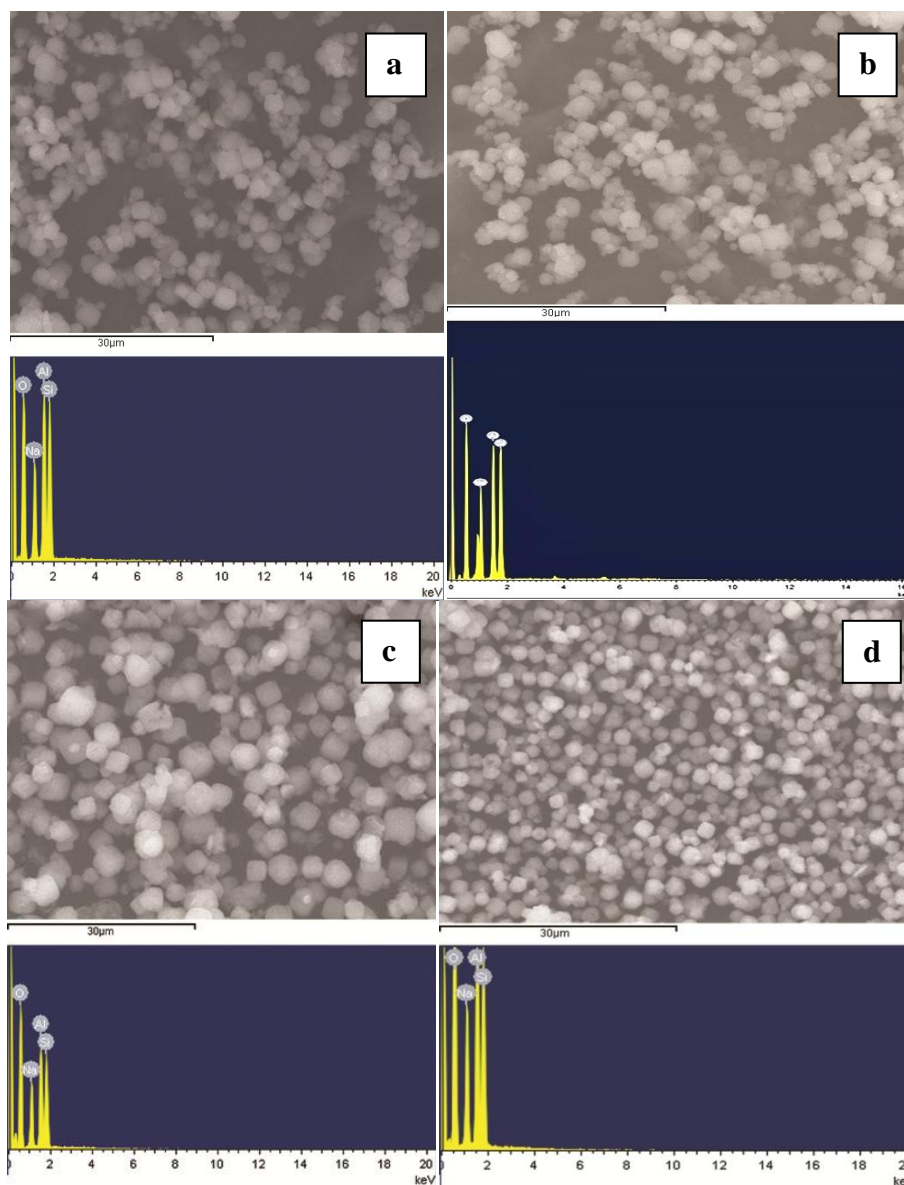


Fig.4. EDAS of (a) raw zeolite A, (b) zeolite A treated with water, (c) zeolite A modified with low PVA and (d) zeolite A modified with high PVA.

Table 2. Elemental Composition of raw, water treated, low and high PVA modified zeolite A.

Elements	Raw Zeolite A		Zeolite A treated with water		Zeolite A modified with low PVA		Zeolite A modified with High PVA	
	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %
O	50.01	62.01	51.50	56.98	55.40	66.81	57.35	68.04
Na	14.03	12.10	14.01	12.47	13.99	11.74	12.95	10.84
Al	16.98	12.49	16.97	14.87	14.82	10.60	14.54	10.36
Si	18.98	13.40	17.52	15.68	15.79	10.85	15.16	10.76
Total	100							

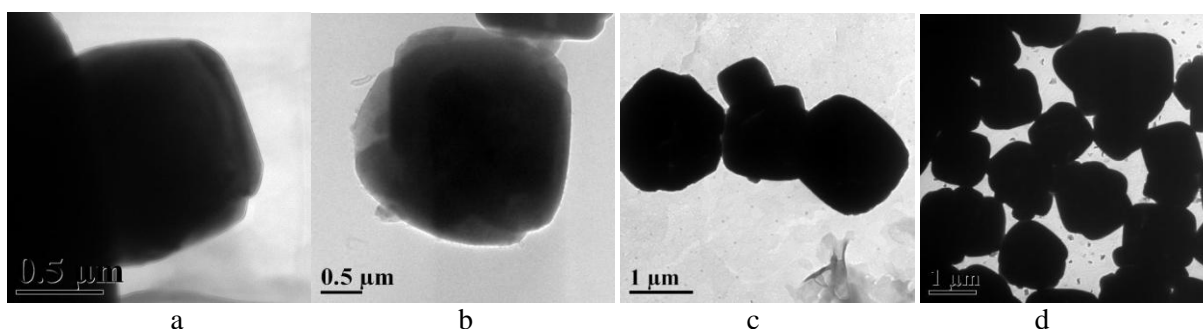


Fig.5. HRTEM micrographs of (a) raw zeolite A, (b) zeolite A treated with water, (c) zeolite A modified with low PVA and (d) zeolite A modified with high PVA.

Particledistributions

The statistical particle size distribution of raw zeolite A (with calcination), zeolite A treated with water (without and with calcination), zeolite A modified with low PVA (without and with calcination) and zeolite A modified with high PVA (without and with calcination) are shown in Fig. 6 (a-g) respectively.

The graph was plotted after statistical image analysis in the frequency and particle size range. It is observed that for raw and water treated zeolite, the distribution of the particles is the highest, nearly within a range of 2.5-3.5 μm, whereas in the case of modification with low and high PVA, there is a reduction of the particle size with narrower distribution. After modification with low and high PVA the maximum particles have a size within the range of 2-2.5 μm (29 % size reduction as compared to raw zeolite A) and 1.5-2 μm (43 % size reduction as compared to raw zeolite A) respectively. This indicates the reduction of the particle size after modification with a high PVA is quite significant. The distribution of the particles is quite uniform as observed from Fig. 6(g). This happens due to a hydrogel surfactant PVA, which helps to stabilize as well as de

agglomerate and disperse the particles during sonication.

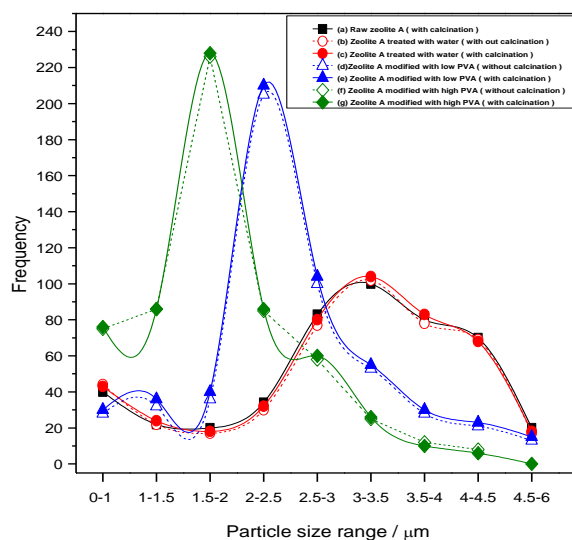


Fig.6. Particle size analysis of (a) raw zeolite A (with calcination), (b) zeolite A treated with water (without calcination), (c) zeolite A treated with water (with calcination), (d) zeolite A modified with low PVA (without calcination), (e) zeolite A modified with low PVA (with calcination), (f) zeolite A modified with high PVA (without calcination) and (g) zeolite A modified with high PVA (with calcination).

From particle size analysis (Fig. 6.), it is found that there was no significant change in the particle size after calcination, which is also correlated with the XRD analysis.

CONCLUSIONS

It is found in the present study that the modification of zeolite A using PVA is possible. As a result of PVA modification, the particle size is reduced about 43 % from 3.5 μm to 1.5 μm with better dispersion. The zeolite particles have changed their shape from cubical to slightly spherical by the effect of calcination. Further research should be focussed on the optimisation of the surface modification to raise the zeolite efficiency for various applications. The modified zeolite A may be used for purification of waste water, soil remediation, as a catalyst, molecular sieve, ion exchanger, adsorbent and for the removal of CO_2 from gas streams.

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ЕФЕКТ ОТ МОДИФИКАЦИЯТА НА ЗЕОЛИТ А С ПОМОЩТА НА ПОЛИВИНИЛ-АЛКОХОЛ (PVA)

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(Резюме)

Извършена е структурна модификация на суров зеолит А при 80°C с ниско- и високомолекулен поливинилов алкохол (PVA). Продуктът е охарактеризиран чрез рентгеноструктурен анализ (XRD), Фуриерова инфрачервена спектроскопия (FTIR), полево емисионна сканираща електронна микроскопия (FESEM), енергийно дисперсионна рентгенова спектроскопия (EDS), трансмисионна електронна микроскопия с висока резолюция (HRTEM) и образен анализатор (IA). XRD показва, че молекулите на PVA се захващат на стените [6 0 0], [6 2 2], [6 4 2], [6 4 4], което води до намаляване на кристалитните размери на модифицирания зеолит. Калицинирането при 600°C не е достатъчно за отстраняването на макромолекулите на PVA от стените на зеолита. FTIR показва понижени нива на шум в спектралните линии в областта 400-420 cm⁻¹ за модифицирания зеолит. Това е индикация за изглаждане на повърхността на суровия зеолит след нанасяне на PVA. Намерено е от FESEM, че формата на модифицираните частици слабо се променя към леко сферични с намаляване на размерите. EDS потвърждава повишаването на съдържанието на кислород, докато съдържанието на Na, Al и Si намаляват след модифицирането. HRTEM показва, че модификацията е значима за зеолити с високомолекулен PVA, а размерът на частиците намалява до 1.5 μm. Анализът на размера на частиците показва, че след модификацията с ниско- и високомолекулен PVA най-много частици са с размери в интервала 2-2.5 μm (29 % намаление спрямо суровия зеолит А) и 1.5-2 μm (43 % намаление спрямо зеолит А) и че няма значимо изменение в размерите след калциниране.