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Phase Transformation of Rice Husk Ash in the synthesis of NaY Zeolite: Effect of ageing in short crystalline duration

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ABSTRACT

Rice husk became a major product of rice-processing industries. Combustion of rice husk (RH) in air produces residues identified as rice husk ash (RHA) with content in SiO₂ that varies from 85-95% based on the burning conditions, the type of furnace and the rice variety. Emphasizing on sustainable consumerism waste, rice husk ash can be used an alternative way to replace the constituent of silica element in zeolite frameworks. RH silica was prepared by burnt at 700°C in 5 h. The zeolite gel was prepared in sodium form (NaY), hydrothermally synthesized using silica source from rice husk. The zeolite gel was prepared with the following batch composition: 3.4Na₂O: Al₂O₃: 9.5 SiO₂: 136H₂O synthesized at various ageing time (8, 12, 24 h) in 12 and 24 h of crystallization reactions. The phase and morphology of the synthesis products were characterized using X-Ray Diffraction (XRD) and field emission scanning electron microscopy (FESEM), respectively. XRD results indicated that phase transformation was occurred, wherein an amorphous phase of RHA has been transformed into crystalline phases. It was also showed that the ageing and crystallite duration affected the NaY zeolite crystallization process. As the crystallite reaction time is increased, the metastable phase was transformed into a more stable phase which is NaP zeolite. Presences of NaP zeolite obviously seen when prolonged of ageing duration up to 24h. Also, NaY zeolite product contained a mixture of NaA zeolite. This study also showed that the images of zeolite produced reflect on the XRD results attained. The optimum crystallization time for the synthesis of zeolite NaY with RHS in modified of simple and easy hydrothermal route was 8h of crystallite reaction with age of 8h.

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INTRODUCTION

Rice husk (RH) becomes one of the most abundance agricultural products in Malaysia. A million tons of rice husks, which can be considered as agricultural waste, are thrown as a waste every year and usually ends up with open burning process, causes environmental pollution and disposal problems. Discovering and recovering high value materials from RHs is becoming a potential research since burning and decomposition of RH will produce a value silica compound. In fact, due to growing environmental concern, and the need to conserve energy and resources, effort have been made to utilize the resultant ash as for a building proposes, semiconductor industry and additive in membrane applications. (Rafiee, E., *et al.*, 2012; Jamalludin, M. R., *et al.*, 2013; Adam, S. F., *et al.*, 2006) Basically, RHA is an active catalyst and a good material for catalyst support due to high surface area (Sun, L., and K. Gong, 2001) also can be used as an alternative cheap source of amorphous silica for zeolite preparation. The extensive usage of RH in the preparation zeolites for separation processes such as sorbent, as well petrochemical processes is widely explored and studied. (M.Chareonpanich *et al.*, 2004)

Basically, zeolites are hydrated crystalline aluminosilicates composed of tetrahedral TO₄ units (T=Si or Al) which are linked together by sharing oxygen atom to form regular intracrystalline cavities and channels of atomic dimensions. (Prasad Dey, K., *et al.*, 2012) Because of molecular sieve properties along with high thermal, mechanical and chemical stabilities, zeolite are extensively used as catalyst, adsorbents, ion exchanger, liquid and gas separation, and membrane. (Bohra, S., *et al.*, 2014) Besides, zeolites are conventionally prepared by the hydrothermal method of gel containing silica, alumina, cation, template and water. (Prasetyoko, D., *et al.*, 2006) Also, silica element can be modified and replaced with different silica sources. Many researchers have

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established the used of RHA silica in different of method and routes to synthesis different zeolites. (Milan, K. N., *et al.*, 2012; Nzaila, E. *et al.*, 2011) By use of RHA as silica source, Khemthong, P., *et al.* 2007 able to synthesized Zeolite LSX by hydrothermal process, while Katsuki, H., *et al.* 2005 obtained ZSM-5 by conventional- and microwave-hydrothermal method. Also, zeolite beta was obtained from rice husk ash by hydrothermal process. (Prasetyoko, D., *et al.*, 2006)

For a better understanding of the relation growth behaviour of the products, an investigation of Y-type formation mechanism is important to look back on the parameter deal with. Theoretically, the formation mechanism of zeolite under hydrothermal condition is very complex. (Wong, W.C. *et al.*, 2001) It included several key parameters such as agitation time, agitation temperature, ageing time, ageing temperature, crystallization time and crystallization temperature on the zeolite preparation. (Htun, M.M. H. *et al.*, 2012), However, the optimization and discussion regarding to ageing time in room temperature emphasized at lower crystallite duration upon emphasizing on sustainable requirement is rarely discussed. Khabuanchalad, S., *et al.*, 2008 has reported that ageing duration did not have a great effect on the procedure of zeolite synthesis. It was contradict with Al-Zaidi, S.B.Y., 2012, reported a deformation of Na-Y is obviously shown with the increased of ageing period. As reported by him, the intensities of peak is obviously seen and the effect of ageing becomes an interest in the mechanism of crystal growth and the results show that the process expected to perform better when gel for a zeolite hydrothermal synthesis is proceed rapidly. (Al Zaidi, S. B. Y., 2012) The investigation showed that there is no chemical transformation of silica upon mixing, in fact, ageing have been found during slowly dissolution of silica, in which releasing silicate anions into the surrounding solution. These silica ions react with aluminate anions to produce aluminosilicate species continuously proposed to the formation of zeolite. (Ginter, D.M., *et al.*, 1992)

On the other hand, temperature and duration effects during hydrothermal reaction on NaY zeolite give a great effect in producing Y zeolite. The crystallization temperature capable to change the zeolitic phase that obtained wherein it can changes its induction time before crystallization; in other words, the increment of temperature implies for reducing of induction period in crystallite process. (Petkowick D.I., *et al.*, 2008) However, many researcher have been studied various temperature that used for the synthesizing of zeolite Y. According to Araújo, L.R.G., *et al.* 1999, synthesized zeolite Y can be found at 196°C using autoclave reactor for 7 days. Bo W. and M., Hongzhu, 1998 reported the zeolite NaY by hydrothermal synthesis, obtained irregular shaped with an average particles sized less than 0.3µm at 100°C with 24 hours crystallization time. Also, Sang, S., *et al.* 2006 reported the synthesis of zeolite Y by hydrothermal technique using two-stage variable temperature was found at higher temperature induced the formation of zeolite P instead of zeolite Y. Although many explanation have been put forward, giving very quality and worthy data. Basically, zeolites Y synthesis was performed in range of 100 to 200°C for periods of time between several hours or weeks.

In this present work, static hydrothermal method is applied for the synthesis of NaY zeolite powders. The intermediate reaction steps for the extraction of silica from RHA into NaY zeolite could be omitted to provide a simple and easy steps, followed by the investigation of aging effect also short duration crystallite reaction. Also, the static hydrothermal has been modified, offering new insight for more simple method. Basically, NaY zeolite synthesized at lower temperature, but, the optimization of ageing duration in room temperature has not be discussed especially by used of RHA as silica source. As a result, economical method by approaching the use of room temperature for ageing reaction was conducted. Also, shorter duration of crystallite reaction carried out in this work. XRD and FESEM were carried out to explain the transformation of RHA into zeolite Y in terms of the effect of ageing and crystalline reaction. 100°C must be strictly controlled as the optimum hydrothermal reaction since lower temperatures are feasible way to synthesis metastable zeolite Y, whereas higher temperatures preferably to synthesize condensed and thermodynamically stable species. (Liu, X., *et al.*, 2003) In fact, implementing lower temperatures also practically in the industries since energy consumption could be reduced.

MATERIAL AND METHOD

Materials:

Rice husk ash, hydrochloride (HCl) (Riedel-de Haën), pellets of sodium hydroxide (NaOH) (Emory), Sodium aluminate (NaAl₂O₃) (Riedel-de Haën), and distilled water were used as the starting materials in the initial mixture for the synthesis of zeolite Y. The silica source used for the experiments was a rice husk obtained from Malaysia rice mill (Jelapang Selatan).

Silica Extraction:

Initially, adhered dirt and soil on RH can be removed by washing with plenty of tap water. Additional cleaning the RH to increase the purity of silica extraction can be performed by immersed RH with hydrochloric acid solution for 24 hour, and again washed with plenty of tap water and later, rinsed with distilled water. The received rice husk was burning at 700°C for 5h in atmosphere, to obtain the silica contain as well as eliminating

the amount of carbon. The yield of silica in this sample ash was 96.45%. Chandrasekhar, S., *et al.* (2006) have stated that the effect of acid treatment, calcination temperature, also rate of heating RH, showed these parameters critically influenced the reactivity and surface area toward lime and brightness of ash. The obtained chemical compositions of rice husk ash are tabulated in Table 1. Also, Fig 1 shows the whole of silica extraction of RH process.

Table 1: Chemical composition of RHA determined by XRF

Chemical composition of RHA	Wt. %
SiO ₂	45
Al ₂ O ₃	0.98
Fe ₂ O ₃	0.24
K ₂ O	1.43
CaO	0.37
C	0.53

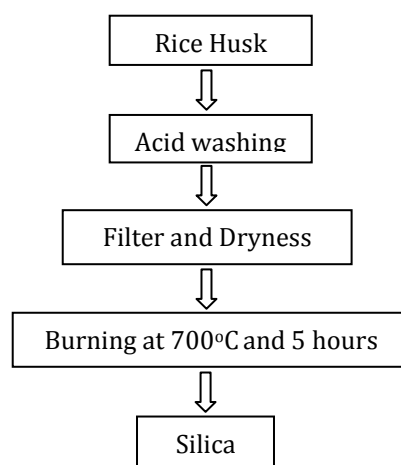


Fig. 1: Flow diagram of the procedure was used to extract silica source from RH

Synthesis of zeolite NaY:

Fig. 2 shows schematically the preparation of zeolite Y powders. In the hydrothermal synthesis, the sodium aluminate solution was prepared by dissolving sodium aluminate powder (1.888g) with NaOH solution (2.361g in 26mL water). Silicate solution prepared by adding RHA (0.18g) into 270 mL water and left for 2hr in stirred condition. The progressive addition of silicate solution and sodium aluminate was carried out with stirring using magnetic stirrer until they dissolved, as to homogenize the reaction gel for 2 hours. The molar composition of the reaction gel was 3.4Na₂O: Al₂O₃: 9.5 SiO₂: 136H₂O adopted from Weitkamp J. and L.,Puppe, 1999. The reaction gel was poured into Teflon-lined stainless steel autoclaves and left undisturbed for ageing as tabulated in Table 2. Then, it was heated at 100°C at autogenic pressure. After hydrothermal reaction, the as-prepared powder were collected by centrifugation (5000rpm) followed by washing with distilled water repeatedly until the washing liquid become almost neutral as observed by the pH meter. The powders were dried at 100°C for 24 hours.

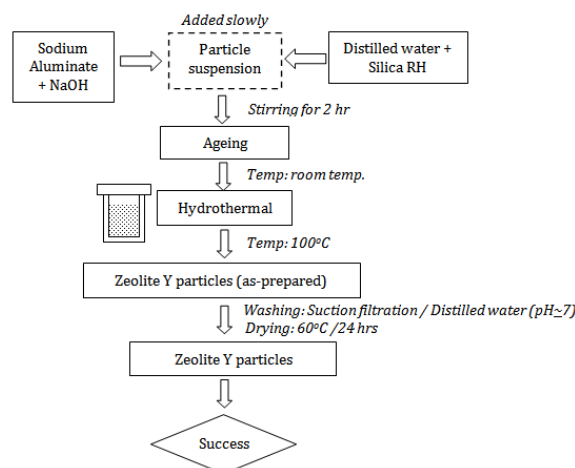


Fig. 2: A schematic representation for the synthesis of Zeolite Y powders using rice husk ash as silica source.

Table 2: Synthesis parameters and their effects on the final products when silica RH was used as the silica source.

Sample code	Ageing time	Crystallization time
A8.8	8	8
A12.8	12	8
A24.8	24	8
A8.12	8	12
A12.12	12	12
A24.12	24	12

Characterizations:

The chemical content of RHA was examined by used of XRF analysis. The XRF test specimens were prepared by pressing fine powder directly into pellets of equal density with the pressures of approximately of 80kN. The crystal phases of the powder were identified using powder diffraction technique by XRD with Ni-filtered Cu-K_a radiation ($\lambda=0.15418\text{nm}$), operating at 40kV and 40mA. The spectrum was recorded over the range $2\theta = 10^\circ$ to 40° with a step size of 0.02° . Phase identification was conducted using JCPDS (Joint Committee on Powder Diffraction Standard) files for zeolites. Furthermore, morphology of the synthesized particles was examined by FESEM operating with an accelerating voltage of 1kV.

RESULT AND DISCUSSION**Characterization of RHA:****The composition analysis of RHA:**

The composition of minerals produced from RHA was shown in Table 1. The gross weight silicon dioxide in RHA was 96.45%, and the residual was carbon and impurities. The conclusion is that preliminary leaching of rice husks with a solution of HCl, has been proved (Ledesma, E.F., *et al.*, 2012) to be an effective way in substantially removing most of the metallic impurities and producing RHS completely white in colour.

XRD analysis:

The micrograph shows the aggregates of particles in RHA. Besides, the diffractograms reveal that RHS in amorphous phase. (Fig 3) It was observe that, only a broad peak with 2θ at 22 degree is a characteristic of the amorphous nature of silica. (Mohamed, R. M., *et al.* 2013)

Scanning Electron Microscopy:

From the FESEM image of silica in Fig 3 (b), amorphous shape which is irregular shape, is a characteristic of amorphous silica was observed.

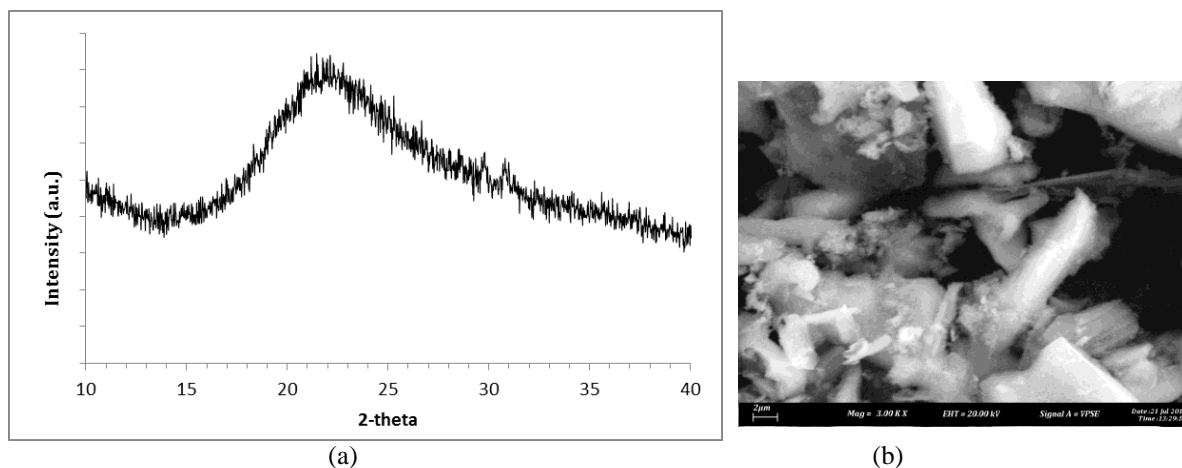
**Fig. 3:** (a) XRD and (b) FESEM image of RHA burned at $600^\circ\text{C}/7\text{ h}$.**XRD analysis of NaY zeolite:**

Fig 5, illustrates the XRD patterns of the synthesized NaY zeolite. The effects of ageing duration for short duration of hydrothermal duration in the formation of NaY zeolite were investigated. All aged sample at 8, 12, and 24 hours were hydrothermally crystallized without stirring at 100°C in 8 and 12 hours as presented in Fig.4 (a) and (b), respectively. It was shown that the diffraction pattern of transformation of silica RHA phase into crystalline phase of zeolite is already occurred at 8 hour of age for both hydrothermal times. The crystalline phase is identified as NaY with JCPDS no. 38-0240, but it is interesting to point out that the mixture of another phase were also be found which are NaA (JCPDS no.39-0222) and NaP (JCPDS no. 39-0219) crystals

especially for the crystalline reaction of 12h. Besides, the broaden peaks for both crystalline reaction gives similar diffraction with in increase of ageing duration.

However, samples aged at 24h in 12h crystallize reaction create a phenomenon. It was obviously illustrated that the transformation of NaA to NaP takes place once the duration of age is extending to 24 hours as can be seen at 2θ : 21.62° . Also, peaks of NaA suddenly weaken and disappear evidently at 2θ : 23.941° , 27.056° , 29.889° . Longer duration of ageing time (24h), creates a metastable of NaY zeolite in which experienced to transform into more thermodynamically stable, which is NaP. (Bohra, S., *et al.*, 2014)(Liu, X., *et al.*, 2003)(Rahman, M.M., *et al.*, 2009) In the same time, longer ageing results in the isolation of the Al-rich regions by further incorporation of Si into the precipitate. This gives rise to larger number of smaller nuclei and results in a higher final yield of NaY. (Ginter, D.M., *et al.*, 1992).

On the contrary, the characteristic peak to the product from 8h of crystalline reaction are different from those are crystallised in 12h. The reaction time of 8h crystallization (Fig 4(a)) produces a minor crystalline phase of NaP along with NaY phase. Thus, it was illustrated that NaA favoured to exist in lower synthesis duration. This situation was reflected on Subotic B. *et al.*, 1982 studied, that all silica previously which is contained in zeolite A is actually built into zeolite P when prolonged reaction of crystalline reaction conducted, despite of the occurrences of metastable of NaY phases.

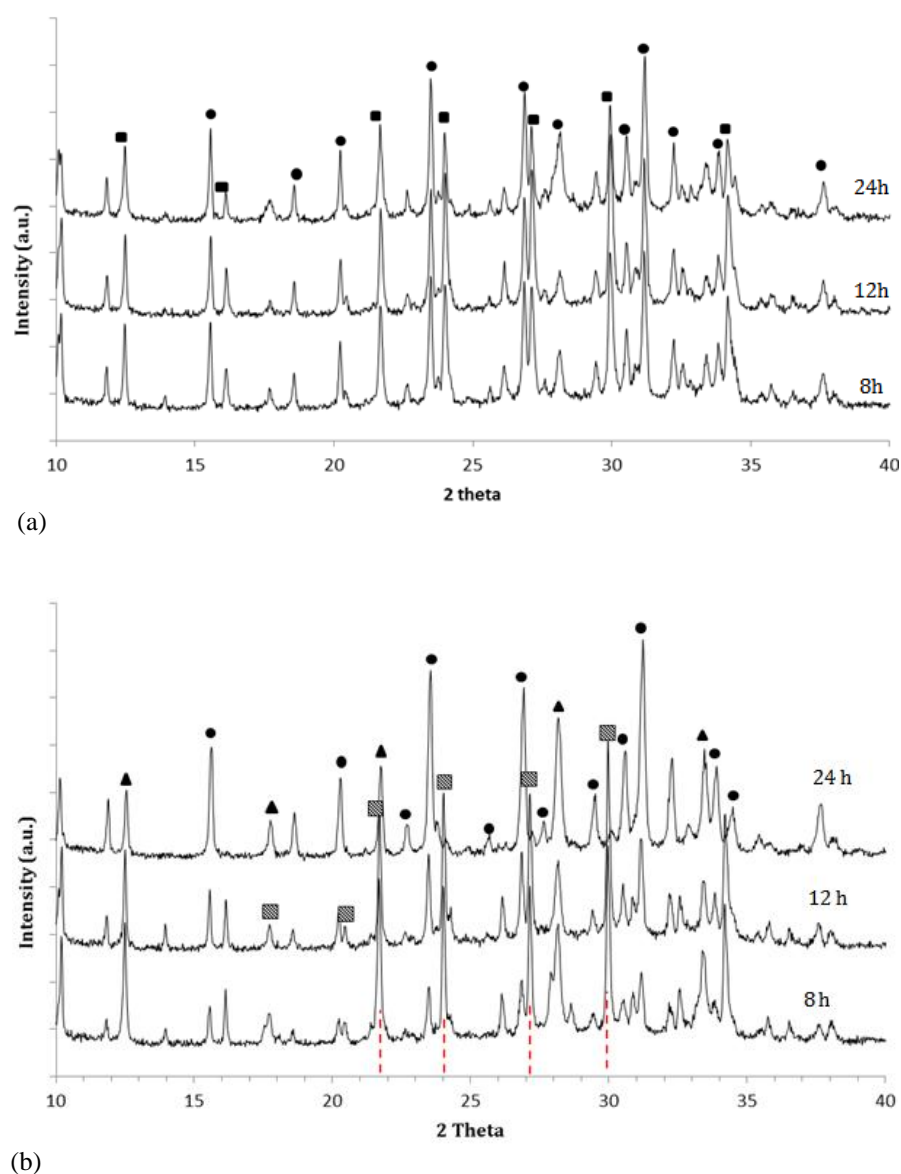


Fig. 4: XRD patterns of (a) 8h and (b) 12 h crystallization duration

●: NaY; ▲: NaP; □: NaA

FESEM micrograph of NaY zeolite:

To corroborate the structure of the product of different ageing duration, FESEM micrograph was conducted. Fig 5 shows the FESEM microstructure of NaY zeolite powders obtained at 8h of crystallite reaction, aged for (a) 8h, (b) 12h, (c) 24 and those are synthesized in 12 h, aged for (d) 8h, (e) 12h, (f)24h. FESEM images show that crystallization time was found to have a substantial effect on the crystal size and morphology. It clearly reveals that 8 h crystallites sample consist of aggregated amorphous aluminosilicate gel. All the gel particles with irregular shapes were observed (Fig 5a). At this moment, the existence of nuclei is actually occurred but, it was wrapped by amorphous mass, (Valtchev, P., and K. N.,Bozhilov, 2004) which is evidently proved by verification of NaY phase in XRD. With further increase in ageing time up to 12h, flower-like and agglomerated cubic shaped morphology for the NaY zeolite was obtained. It is worth to mentioned that, initially (12h of ageing time) flower like NaY zeolite was formed surrounding with agglomerated cubic shaped which could be act as nucleating sites for particles growth. Similar result has been found by Bohra, S., *et al.*, 2014, claimed that flower-like particles of faujasite zeolite could be coexisting with the existences of some amorphous particle. Once the reaction time reach to 24h, the overlap of cubic crystals were obviously obtained, present a sign of the presences of NaA zeolite. Besides, other crystals without defined shape were observed which is probably related to sodalite phase. (Alves J. A. B. L. R. *et al.*, 2014) Also, ageing up to 24 h, formation of nuclei from crystals was subsequently grows.

For the crystallite reaction of 12h, overlap cubic in agglomerated morphology was obtained. The micrographs shown in Fig 5d confirm the formation of zeolite A (presence of cubic crystals) as previously discussed in XRD. As the ageing reaction was increased, a large number of loosely pack zeolite crystals were produced in smaller size. The particles exhibited in irregular crystals shape and it was found that cubic particles shape apparently less comparing to 8 h of crystallite reaction as evidenced by XRD test. In fact, the analysis of FESEM lead to the conclusion that transformation of zeolite A into zeolite P phenomenon took placed by nucleation and crystal growth from the solution as previously highlighted in XRD discussion.

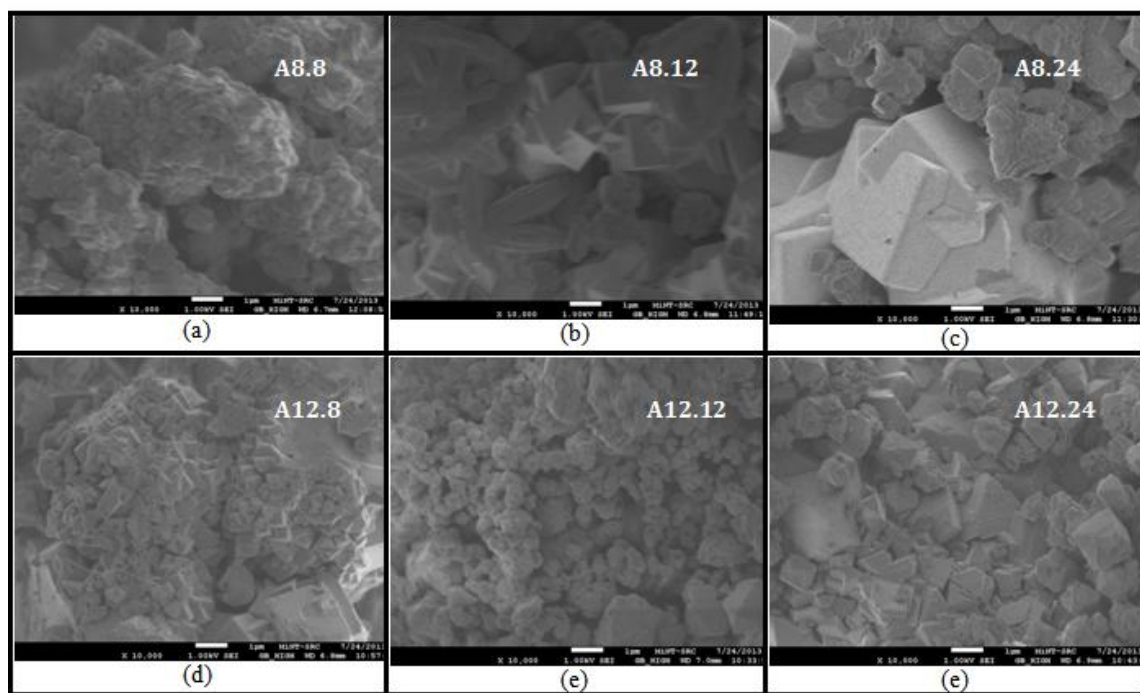


Fig. 5: FESEM microstructure of zeolites produced.

Conclusion:

This work reports an experimental study on short crystalline duration, varied in room temperature ageing duration by developing simple and easy step of hydrothermal reaction. Synthesis parameters are seen to have significant effects on the zeolite produced and crystals formed at the end. XRD results revealed that, the crystallization time of 8h, zeolite Y could be observed with mixture of NaA zeolite through this route. Further longer the ageing duration, crystals are able to produce which is apparently clear shown in FESEM. Besides, 12 h of crystallization reaction also illustrated similar trend at lower ageing duration.(8 and 12h) However, up to 24h of age duration, it was noticed that obviously competitions among NaY, NaA and NaP zeolites was occurred during the synthesis reaction. This is because of NaA and NaY are the metastable pattern while NaP is relative stable one. Thus, by extending the ageing duration, it was negatively for the synthesis process since

tendency of NaY zeolite to achieve the stable phase giving more actively. The optimum crystallization time for the synthesis of zeolite NaY with RHS was 12h of crystallite reaction with 8h for ageing duration.

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