I-GT-058: Synthesis of Green Palm Oil Mill Fuel Ash Zeolites (G-POMFAZ) Using Hydrothermal-Alkaline-Ultrasound Technique (HAUT)

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Abstract

The tremendous fuel ashes are deserted by palm oil industries into environment were not processed for commercializable products such as zeolites, etc. There are a few methods for zeolites synthesis, such as molten-salt, sol-gel, etc., but it takes considerable time, extensive set up and needs to high cost, thus this work aims to solve this issues by knowledge transfer of hydrothermal-alkaline-ultrasound technique (HAUT) for palm oil mill fuel ash (POMFA) based zeolites synthesis as sustainability and green material processing technology initiatives. The knowledge transfer methods of the POMFA characterization and analysis procedure completed via talks, workshop, practical, exercise and data analysis. The analysis of POMFA using Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), Brunauer-Emmett-Teller (BET), Fourier Transformed Infra Red (FTIR), and X-Ray Flourescent (XRF) for composition approval has been conducted. The XRF result reveals 37.92 % silicon dioxide and 3.42% aluminium oxide etc. as major components typically used in the manufactures of zeolites. Next, the green technology transfer for zeolites synthesis from POMFA via HAUT utilization, process parameter effect, analysis equipments and operational procedure has been achieved. The highest Green Palm Oil Mill Fuel Ash Zeolites (G-POMFAZ) using HAUT was found at the operation condition of POMFA-akaline ratio of 1:2, ultrasonic exposure time of 2.5 h, medium temperature of 80°C, irradiation power of 800 W and frequency of 45 kHz. The synthesized G-POMFAZ can be considered as an alternative material for industrial application, like construction, petrochemical, etc. The knowledge transfer program via training and dissemination of morphology analysis of G-POMFAZ was directed to the company staff. The staff knowledge is drastically increased on the POMFA characterization and zeolites synthesis. The project contributes also to the reduction of the CH Biotech-wastes and contaminated particulate matter gradually.

Keywords: *G-POMFAZ, Characterization, HAUT, Morphology Analysis, Technology Transfer*

1. Introduction

Malaysia is currently one of the world leading producers and exporters of palm oil such as CH Biotech Sdn Bhd, etc. In year 2007 up to 2011, production of crude palm oil increased from 15.8 to 19.8 million tonnes, and the palm oil export of 36.75 % in year 2011. The export earnings of oil palm products were RM 45.1 billion that approximately USD 13.1 billion (Ismail, *et al.*, 2013; Awal, *et al.*, 2015). Commonly, for each bunch of the fresh palm fruit consists of 21% of palm oil, 6–7% of palm kernels, 14–15% of palm fibers, 6–7% of palm shells and 23% of empty fruit bunches can be produced. Besides the production of crude palm oil, a large amount of solid waste such as palm fiber, shells, and empty fruit bunches is also produced as residual output from the palm oil industry. Typically, solid wastes from palm oil residue are used as a fuel to produce steam for electricity generation in order to generate energy. After combustion, an ash is estimated about 5% by weight of solid waste (Foo & Hameed, 2009). The solid waste in the form of Palm Oil Fuel Mill Fuel Ash (POMFA) is by-product from burning process in palm oil plantation, like CH Biotech Sdn Bhd, which palm nut and fiber of palm are burnt at temperature of about 800-1000^{o}C. The POMFA is characterized by a spongy and porous structure in nature, of which it's main components in the angular and irregular form, with a sizable fraction showing cellular textures. Meanwhile, raw palm ash contains a rather spherical particle with a

median size of 183.0 µm, and small particles ground palm ash were individually crashed shape structures with a median of 15.9 µm and 7.4 µm. The approximation value of oil palm ash production in Malaysia is predicted at 4 million tons per year. Usually, the POMFA would be disposed off for landfills because of the limited uses and also for agricultural application. This situation could lead to pollution case in the future. One excellent way to solve this potential problem, many researchers have studied the utilization of POMFA uses in concrete admixtures, catalyst, cement replacement material, concrete additive, aqueous and gaseous pollutant-removing adsorbents (Chong, *et al.*, 2009; Chandara, *et al.*, 2010; Ho, *et al.*, 2012; Yusuf, *et al.*, 2015). Previous results from researchers have shown that these oil palm ashes contained a high amount of silica in amorphous form. The POMFA can also be utilized for toxic gas removal (SO_x). The active compound (silica, alumina, potassium, calcium and hidrated water) in the adsorbent prepared from POMFA is believed to be responsible for the gas and wastewater contaminants separation. Moreover, the feasibility of using the shell and fiber ash as a construction material is also applicable. The preliminary research about the possibility of POMFA utilization for adsorbent such as zeolites production has been has been reported, but the process condition of feasible parameter, methodology and economic analysis are in progress. The main reason for the wide use of zeolites is their unique physical and chemical properties including large surface area, sieve-type molecular structure and high adsorption capacity. The zeolites can also be manufactured from another agricultural wastes, like sugar cane bagasse, etc. (Yeliz, 2010; Moises, *et al.*, 2013).

Based on the above reasons, POMFA as solid waste containing silica which has great similarities with the raw materials typically used in the manufacture of zeolites. It could be hypothesized that POMFA has a big possibility for adrsorbent production such as zeolites. The extraction of zeolites is more expensive and causes considerable degradation to the environment. Therefore, the synthesis of zeolites from POMFA using HAUT is a viable program, and in this KTP program, the involved industry is CH Biotech Sdn Bhd.

2. Methodology

The synthesis of zeolites from POMFA involves 3 main phases:

Phase I: Characterization and analysis of POMFA as zeolites feedstock.

Phase II: POMFA utilization for zeolites synthesis using HAUT.

Phase III: Pattern, morphology and composition analysis of synthesized zeolites.

2.1. Phase I: Characterization and analysis of POMFA as zeolites feedstock.

The understanding of characterization and analysis of POMFA were carried out by the following steps, namely: a. knowledge transfer and training on the POMFA characterization and analysis equipments; b. knowledge transfer and training on the POMFA characterization and analysis procedure.

2.2. Phase II: POMFA utilization for zeolites synthesis using HAUT

The realization of POMFA utilization for zeolites synthesis using HAUT was conducted as follows: a. knowledge transfer and training on the feasible raw materials of POMFA for zeolites synthesis; b. knowledge transfer and training on the HAUT for zeolites synthesis; c. knowledge transfer and training on the process parameter effect on the zeolites synthesis.

The knowledge and technique of zeolites synthesis will be disseminated through the alkaline hydrothermal methods. Characterization and analysis of feedstock, synthesized zeolites and several process parameters like temperature, time and alkaline/POMFA ratio was observed .The work procedure is described as follows: The raw POMFA was obtained from an oil palm mill at Hutan Melintang in the state of Perak, Malaysia. The collected POMFA has to be sieved through a sieve No. 16 (1.18 mm opening) in order to remove foreign materials and uncombusted palm fiber. The residue ash retained on a sieve is about 10% by weight. To improve reactivity, grinding process of POMFA is needed in order to improve the pozzolanic reaction.

HAUT was used in order to produce zeolites from POMFA. The required alkaline method involves a few steps. Firsty, the mixture of POMFA and potassium hydroxide (KOH) as pre-determined ratio were milled and fused in a furnace at the different temperature and time. Secondly, the POMFA to alkaline ratio was varied by weight and the ratio value of 1:1, 1:2, 1:3, 1:4 and 1:5, and the POMFA particle size was fixed at 800 µm. Thirdly, the resultant fused mixture must be cooled at room temperature and was further crushed in order to obtain the homogenous mixture, and the water of 100 mL was added to the mixture. Fourthly, the mixture was treated by ultrasound irradiation with frequency of 45 kHz and power of 400 W, 500 W, 600 W, 700 W and 800 W, exposure time of 30 min.,

60 min., 90 min., 120 min. and 150 min., hydrothermal medium temperature of water bath was 40°C , 50°C, 60°C, 70°C and 80°C. The fine slurry was agitated and shaked in a water bath for 24 hours. The mixture was washed with water until pH reached almost neutral and the filtrat was recovered using filter paper, and it was dried in oven at 110°C for 2 hours. The precipitate was then repeatedly washed with distilled water to remove excess KOH, followed with filtration and drying process in order to obtain the zeolites. Finally, the resulted zeolites were analyzed using XRD, SEM and XRF.

2.3 Phase III: Pattern, morphology and composition analysis of synthesized zeolites.

The information of pattern, morphology and composition analysis of synthesized zeolites were disseminated through the following ways, specifically: a. knowledge transfer and training on the pattern analysis of synthesized zeolites; b. knowledge transfer and training on the morphology analysis of synthesized zeolites; knowledge transfer and training on the composition analysis of synthesized zeolites.

3. Results and Discussion

3.1. POMFA characterization and analysis

The POMFA characterizataion and analysis have been demonstrated. The result of Scanning Microscopic Analysis (SEM) is shown in *Figure* 1.

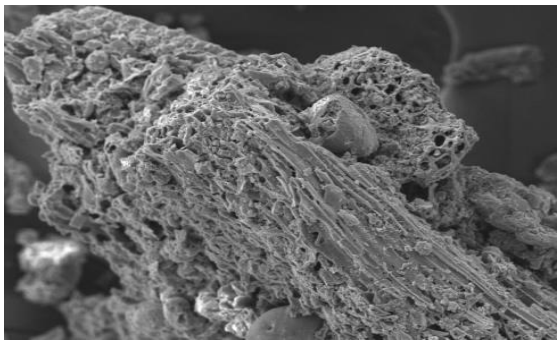

Figure 1: SEM Micrograph of raw POMFA.

The SEM image of raw POMFA shows more cubic structure than fibrous form. The porous characteristics of the particle ash has been identified by SEM. The morphology shows the irregular form with a sizable fractions of cellular textures. The POMFA has also covered by an aluminosilicate glass phase (Foo and Hameed, 2009). Next, the XRF analysis of the raw POMFA can be divided into major and minor chemical constituents. The constituents of POMFA are SiO_2 (37.92%), CaO (6.21%), K_2O (5.35%), Al_2O_3 (3.42%), Fe_2O_3 (3.19%), P_2O_5 (2.50%), MgO (1.76%), SO_3 (0.73%), Cl (0.38%), TiO_2 (0.27%), ZrO_2 (0.09%), MnO (0.08%), Na_2O (0.04%), CuO (0.03%), SrO (0.02%), Rb_2O (0.02%), ZnO (0.01%), Cr_2O_3 (0.01%), V_2O_5 (12.39%), NiO (11.34%), Nb_2O_5 (8.97%) and As_2O_3 (5.27%). The POMFA containing SiO_2, Al_2O_3, CaO, etc. was also reported by researchers (Sata *et al.*, 2010; Aziz *et al.*, 2014; Salih, *et al.*, 2015), but the difference in chemical composition may be caused by various operating systems in palm oil mills and raw material sources. The sources of used POMFA reveals BET surface area of 20.11 m^2/g.

The raw POMFA has been analyzed by XRD. The XRD pattern of raw POMFA analysis is displayed in *Figure* 2. which indicated that it consists mainly of amorphous aluminosilicate glass, quartz and mullite.

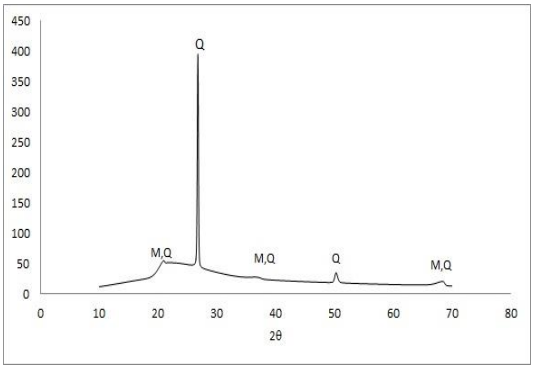

Figure 2: XRD pattern of raw POMFA.

These patterns are almost similar with fly ash. Broad hump around 20°-22° (Bohra, *et al.*, , 2014) and 15°-40° (Salih *et al.*, 2015) indicates the amorphous nature of POMFA.

FTIR analysis of POMFA is exhibited in the *Figure* 3.

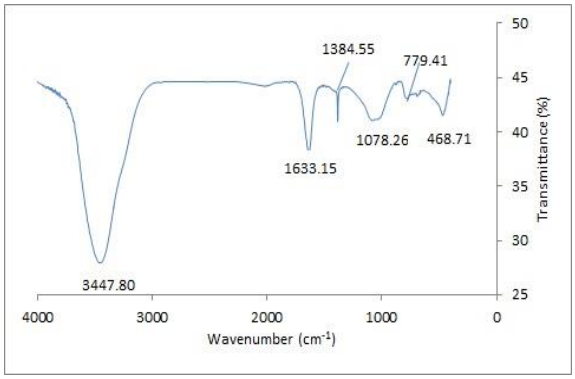

Figure 3:. FTIR of raw POMFA.

Figure 3 shows the characteristics absorption band of raw POMFA. Stretch band at 3447.8 cm^{-1} belongs to Silanol group, Si-OH. This band is reported in range of 3200-3750 cm^{-1}. The sharp peak at 1384.55 cm^{-1} and broad peak at 1078.26 cm^{-1} represents C-O stretching band, alcohol and phenol (Allwar, 2012). The unique characteristic of POMFA is also reported around 1300-1400 cm^{-1} that belongs to alcohol and phenol on adsorbent surfaces. Al_2O_3 presence was also indicated by the absorption band at 779.41 cm^{-1} (Najib *et al.*, 2009). FTIR result shows POMFA contained silica and alumina that justify the XRF compositions obtained.

3.2. Process parameter influence on the zeolites synthesis using HAUT

The influence of process parameters on the formation of crystalline zeolites was observed via experiments. It was focused on the ratio of POMFA-KOH, ultrasound exposure time, medium temperature and irradiation power.

3.2.1. POMFA-KOH ratio.

The ratio of POMFA-KOH plays on the zeolites synthesis an important role. The role of POMFA-KOH ratio on the formatted zeolites is shown at *Figure* 4 and Table 1.

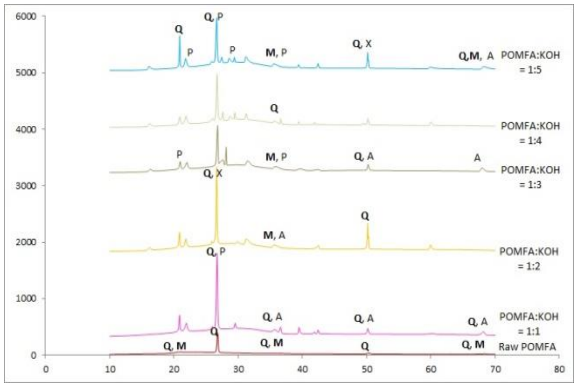

Figure 4: Diffraction patterns of synthesized zeolites via various ratio of POMFA-KOH (T=80°C, P=800 W, f= 45 kHZ and t=2.5h,).

Table 1: Major peaks intensity of synthesized zeolites via various ratio of POMFA-KOH.

Angle of Incidence (°)	Intensity of major peaks				
	POMFA-KOH ratio				
	1:1	1:2	1:3	1:4	1:5
21	211	198	98	96	438
22	103	108	81	97	100
27	962	1078	71	592	554
50	81	418	90	102	240
Total	1357	1802	340	887	1332

Figure 4 shows the effect of POMFA-KOH ratio on the diffraction pattern of the indicated zeolites, mainly A, P and X for difference framework of zeolites. More formed zeolites if the increasing of POMFA-KOH compared decreasing of the POMFA-KOH ratio. These phenomenons is caused by the dissolution acceleration of quartz and mullite towards zeolite formation. By increasing of POMFA-KOH ratio up to 3 folds for POMFA left quartz mostly unreacted towards formation of zeolite and enhance quartz and mullite. The diffraction patterns also indicate samples feature of the typical peaks around 2θ of 21°, 22°,27° and 50°, that is used for determination of intensity total. The total intensity sum of various KOH is summarized in the above Table 1. The table 1 indicates the highest intensity values at the POMFA-KOH ratio of 1:2. It gives more crystalline effect than others. Otherwise, the POMFA-KOH ratio of 1:5 produces more peaks of resulted zeolites but it causes more quartz and mullite peaks. Total addition of major peaks intensity endorses a semi-quantitative index of crystallinity from powder diffraction. The best quality is determined by the higher X-ray intensity (Shams & Ahi, 2013). The POMFA-KOH ratio of 1:2 also reveals more cubic structure compared the others.

3.2.2. Ultrasound exposure time

The ultrasound exposure time effect on the resulted zeolites is reflected at *Figure* 5 and Table 2.

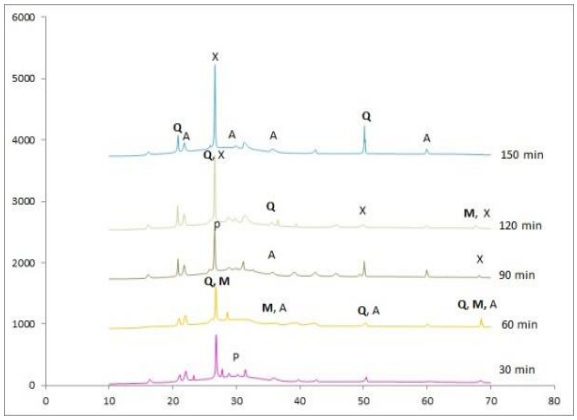

Figure 5: Diffraction model of synthesized zeolites via various time (POMFA-KOH=1:2, T=80°C, P=800 W, f= 45 kHZ).

Figure 5 illustrates the influence of ultrasound exposure time on the diffraction impression of the produced zeolites. At the irradiation time 30 min., low intense of zeolite A and P can be seen and the most intense peak is still quartz and mullite. The highest and more peaks of quartz and mullite are represented by the zeolites at 60 min. The ultrasound exposure time is extended up to 90 min., the highest intensity is

revealed by zeolites P and X. It appears at 50° and 68° 2θ. The zeolites X as quartz can be seen at the highest peak of 36° 2θ. The longest irradiation time of 150 min. approves the zeolite X (framework structure of X) as the most intense peak with zeolite A (structure of A) dominated. Above 90 min. of the ultrasound treatment, the zeolite X appears as new zeolites phase. The total intensity of various time is given in the Table 2 below.

Table 2: Major peaks intensity of synthesized zeolites via various ultrasound exposure time.

Angle of Incidence (°)	Intensity of major peaks				
	Ultrasound exposure time, min.				
	30	60	90	120	150
21	73	78	212	255	198
22	108	107	119	138	108
27	548	385	482	805	1078
50	58	39	221	26	418
Total	787	609	1034	1224	1802

The obtained data indicates the highest intensity values at the time of 150 min. (2.5 h). The peak intensity of the synthesized zeolite increases with crystallization time. The ultrasound exposure that is complemented with another reviews and investigations (Pal, *et al.*, 2013; Askari, *et al.*, 2013; Azizi & Asemi, 2014). The highest sum of intensity for ultrasound exposure time is also 150 min. (2.5 h).

3.2.3. Irradiation power

The irradiation power impact on the manufactured zeolites is demonstrated at *Figure* 6 and Table 3.

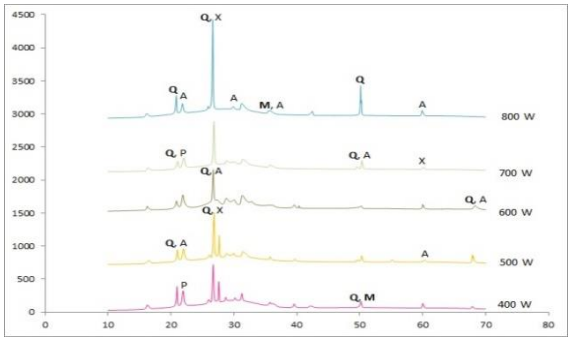

Figure 6:. Diffraction ornament of synthesized zeolites via various irradiation power (T=80°C, POMFA-KOH=1:2, f= 45 kHZ and t=2.5h).

Figure 6 shows the effect of power elevation to the zeolite synthesis with reference to XRD pattern. At 400 W irradiation powers, the mixtures of zeolites A, P (framework structure of zeolites) and X are formed, and the highest peak and intensity are known as zeolites X. Next, the zeolite A becomes major phases at 500 W. Beside that, the zeolite A appears as the most intense and domination at 600 W, and the P and X are as trace zeolites. The zeolite X becomes the most intense peak and the mixtures of zeolite P and A are also formed at 600 W. The total intensity sum of various irradiation power is outlined in the following Table 3.

Table 3. Major peaks intensity of synthesized zeolites via various irradiation power.

Angle of Incidence (°)	Intensity of major peaks				
	Irradiation power, W				
	400	500	600	700	800
21	233.36	128.66	80.26	75.51	198
22	160.32	125.47	139.23	105.71	108
27	407.18	473.67	341.34	471.17	1078
50	75.4	79.22	26.25	101.98	418
Total	876.16	807.02	587.08	754.37	1802

The given data arranges the intensity in XRD powdered pattern with the the highest sum at 800 W. The increasing of ultrasound power influences on the crystallinity of formed zeolites. The ultrasound produces cavitation bubbles with high temperatures and pressures, it results extraordinary heating and cooling rates that develop crystal nuclei. The new formed nuclei overlappes with the presented nuclei that causes the crystal in short time, and the uniform growth of zeolites can be produced (Belviso *et al.*, 2011). Using all power levels, the zeolites X can be formed and becomes the highest intensity.

3.2.4. Medium temperature

The medium temperature effects on the zeolites is reflected at *Figure* 7 and Table 4. *Figure* 7 shows the specimen of synthesized zeolites at temperature 40°C-80°C. At 40°C, most of formed zeolites are as type A with the zeolites P is the highest intensity. The

increasing of temperature up to 50 °C, the zeolites P appears at 22° 2θ, and the zeolite X takes place at 50° and 60° 2θ. The best condition for operation temperature is 80°C.

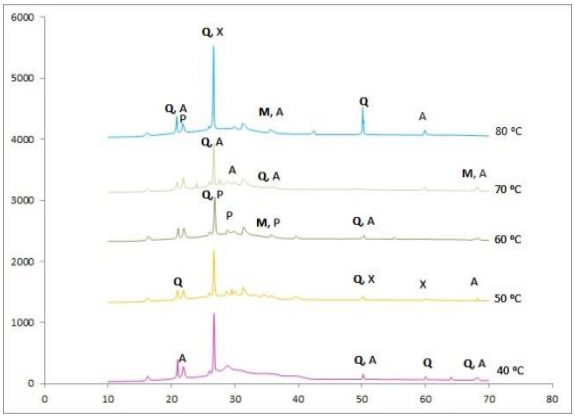

Figure 7:. Diffraction specimen of synthesized zeolites via various medium temperature (POMFA-KOH=1:2, P=800 W, f= 45 kHZ and t=2.5h).

Angle of Incidence (°)	Intensity of major peaks				
	Medium temperature, °C				
	40	50	60	70	80
21	230	110	121	91	198
22	128	97	113	133	108
27	674	559	431	488	1078
50	71	45	44	7	418
Total	1103	811	709	719	1802

The total intensity sum of synthesized zeolites at various temperature is tabulated in Table 4.

Table 4: Major peaks intensity of synthesized zeolites via various medium temperature.

The obtained data display almost pure zeolite phase , but a few total intensities are lower. It can be seen at temperature of 60 °C and 70 °C. The P as zeolites trace dominates phases at 60°C, and the zeolites A is the major phase at 70°C. The zeolites X appears with the highest intensity and temperature, and the zeolite A and P are also formed. Belviso, et al. (2013) report that the zeolites X are found at 45°C and 60°C, so it can be approved by this investigation for majority of obtained zeolites X at 50°C and 80°C. It can be stated that the increasing of temperature can help for rapid crystallization of zeolites.

3.3. G-POMFAZ morphology analysis

The resulted G-POMFAZ is identified by morphology analysis using SEM. The SEM images is presented at *Figure* 8.

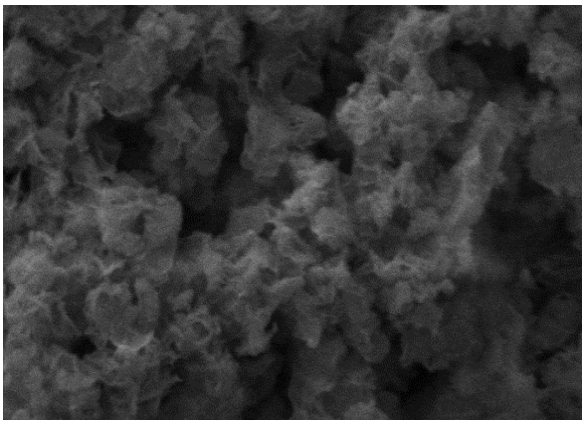

Figure 8. G-POMFAZ SEM images (POMFA-KOH=1:2, T= 80°C, P=800 W, f= 45 kHZ and t=2.5h).

The SEM micrograph displays no spherical particles in zeolites. It indicates that the high conversion of POMFA to G-POMFAZ has been endorsed. The POMPA becomes rough and porous form, the crystalline G-POMFAZ is deposited on the surface of POMFA particles. The difference morphology of resulted G-POMFAZ compared raw POMFA has been approved by using HAUT.

4. Summary

The knowledge about hydrothermal-alkaline-ultrasound-technique (HAUT) for green palm oil mill fuel ash zeolites (G-POMFAZ) has been being transferred for CH Biotech Sdn Bhd staffs. The palm oil mill fuel ash (POMFA) characterization and analysis using SEM, BET, XRD, FTIR and XRF including theoretical and practical exercises have been totally disseminated. The XRF result shows 37.92 % silicon dioxide, 6.21% calcium oxide, 5.35% potassium oxide, 3.42% aluminium oxide etc. as major components. The highest percentage of silicon dioxide, etc. approves the potential raw material of CH Biotech Sdn Bhd waste for G-POMFAZ synthesis. Next, the green technology transfer for zeolites synthesis from POMFA via analysis equipments, operational procedure, HAUT utilization, process parameter effects was achieved. Furthermore, the program of

knowledge transfer via training and dissemination of resulted G-POMFAZ morphology and composition analysis has been executed. The phases of obtained zeolites are not fully pure. The quartz and mullite phases are still found. Further program may include G-POMFAZ purification techniques.

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