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Synthesis and characterization of zeolite-Y using *Ficus exasperata* leaf: A preliminary study

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ABSTRACT

In this study, *Ficus exasperata (Fe)* leaf (sand paper leaf) raw sample was characterized using proximate and ultimate analysis and the ash was characterized using X-ray fluorescence (XRF), X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Fourier Transform Infrared (FTIR) spectroscopy. XRF analysis showed that Alumina (Al_2O_3) and Silica (SiO_2) were 6.50% and 67.50%, Energy Dispersive X-ray (EDX) analysis showed high presence of silica (42.40%), alumina (15.00%) and Oxygen (20.80%). FTIR unveiled peaks with zeolite-Y patterns. SEM analysis indicates good surface morphology and hexagonal shaped crystal lattice in comparison with commercial zeolite-Y.

1. Introduction

Zeolites are aluminosilicates group materials that have pores, crystalline structure and microporous with alkaline earth metals. SiO_4 and AlO_4 tetrahedral are the frameworks composition to form different open structures which are similar to zeolite type MFI (ZSM-5) and ZSM-11 zeolites, which are widely used as catalysts, adsorbents, pH balance and ion exchangers [1–4]. Interestingly, these compounds are significantly efficient as catalysts in chemical services [5]. Studies have shown that the framework structure with 235 types series has been approved with International Zeolite Association (IZA). St. Claire-Deville carried out zeolite synthesis in 1862 and in 1948, Barrer's pioneering work demonstrated that aluminosilicate gels are the sources of a wide range zeolites synthesis [6–10]. Considering the homogeneity of chemical constituents, higher stability and higher activity in catalytic fluid cracking [11,12], synthetic zeolites are used commercially more than naturally synthesized zeolites. Preparations using silica and alumina as a source are often expensive with extreme waste and release of unpleasant smell to the surrounding. Preparation of nano-crystalline zeolites catalyst has been the worldwide focus in order to replace conventional catalysts due to higher stability, pronounced potential in many fields and higher activity in many applications, such as in fluid catalytic cracking [13]. However, researchers have explored the use of local materials and agricultural residues as sources to extract silica and alumina for the production of zeolite due to their low cost of production and eco-friendliness [14,15]. Alternatively, isolation of agro-residues, including *Ficus exasperata* leaf can be used as an aluminosilicate source for zeolite synthesis. Cordeiro et al. [16] characterized bagasse ash and reported that its iron oxide, alumina and silica contents were 3.610, 8.550 and 78.340%, respectively. Amin [17] reported lime, iron oxide, alumina and silica of 2.54, 4.90, 3.60 and 87.40%, respectively. Srinivasan and Sathiya [18] also characterized bagasse ash reported that its iron oxide, alumina and silica contents of 3.61, 8.55 and 78.34%, respectively. Maldonado-Bandala et al. [19] reported silica, alumina, iron oxide and lime contents of 70.5, 5.23, 3.24 and 4.19%, respectively while Gandhi [20] reported 60.26, 10.73, 5.03 and 8.35%. Furthermore, the work of Muazu [21] on bagasse ash characterization showed the percentage compositions of silica, alumina, iron oxide and lime were 57.95, 8.23, 3.96 and 4.52%, respectively.

Ficus exasperata trees are usually about 20 m high with smooth grey bark and very rough leaves, and grow in lowlands and mountains. They are widely used in traditional medicine and palm oil production in Africa. The trees also have several other local uses; the leaves being employed as sandpaper; the plant has some insect repellent properties. *Ficus exasperata* has been used to provide shade in plantations and is planted as an avenue shade tree. In this work, *Ficus exasperata* leaf ash was characterized by X-ray fluorescence (XRF), X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Fourier Transform Infrared (FTIR)

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Table 1
Proximate and ultimate analysis of raw *Ficus exasperata* leaf.

Raw sample	Proximate analysis (wt.%)				Ultimate analysis (wt.%)				
	Moisture Content	Volatile Content	Fixed Carbon Content	Ash Content	C	H	N	O	S
Ficus exasperata leaf	9.00	74.45	14.60	1.95	48.50	6.70	0.20	44.59	0.01

Table 2
Masses and percentage of bagasse before and after burning.

Temperature	M_1(g)	M_2(g)	M_3(g)	%Ash	%Loss
650 °C	500	80.350	419.65	16.07	83.93
650 °C	500	81.025	418.975	16.205	83.795
650 °C	500	80.985	419.015	16.197	83.803
650 °C	500	82.010	417.990	16.402	83.598
Average	**500**	**81.093**	**418.908**	**16.219**	**83.782**

Table 3
Quantitative XRF analysis data of Ficus exasperata leaf ash.

Chemical constituents	X-ray fluorescence analysis (XRF) in wt.%
Na_2O	0.12
MgO	3.15
Al_2O_3	6.50
SiO_2	67.50
P_2O_5	3.70
K_2O	4.45
CaO	5.60
TiO_2	0.56
Fe_2O_3	5.70
Total	97.28
SiO_2/Al_2O_3 ratio	10:1

spectroscopy to determine the components, compounds and novel zeolite –Y synthesis potential for upgrading bio-fuel products.

2. Material and method

2.1. Material

The *Ficus exasperata* (Fe) leaves (sandpaper leaf) were obtained from Ota city, Ado-Odo Local Government Area, Ogun state, Nigeria. The leaves were ground into smaller particles and air dried for 3 days before further studies. The dried sample was characterized with its moisture, volatile matter, ash and fixed carbon contents, as determined by proximate analysis; and carbon, hydrogen, nitrogen, sulphur and oxygen contents, as determined by ultimate analysis, using the CHNS analyzer (Leco CHN 2000 elemental analyzer) according to ASTM D1102 and ASTM D5291 [22,23], as shown in Table 1.

2.2. Preparation of Zeolite-Y

Gel formation, aging and crystallization are the processes involved in zeolites synthesis from *Ficus exasperata* leaves. The leaves were washed thoroughly with deionized water and then air dried for 3 days. The dried leaves were ground with a mill, weighed and calcined at 650 °C for 4 hours to ash according to ASTM D1102- 84 and the masses of the ground *Ficus exasperata* leaves used (M_1) and the ash remaining (M_2) after burning were determined using an electric weighing balance, and mass loss (M_3) as well as percentage mass loss were calculated [24,25]. The calcination experiment was performed four times and the average values of M_1, M_2, M_3, ash and mass loss percentages were determined.

2.3. Characterization

The chemical composition of the sample was determined using Shimadzu XRF-1800 apparatus (XRF, Shimadzu, Japan). The crystallinity phase of the obtained sample was investigated by X-ray diffractometer for X-Ray Diffraction analysis (XRD). The structural bonds were analyzed using Fourier Transform Infrared Spectroscopy (FTIR) and surface morphology was analyzed by Scanning Electron Microscopy (SEM).

3. Results and discussion

Table 2 shows the masses of the ground *Ficus exasperata* leaves (M_1), ash (M_2), loss (M_3), ash percentage and percentage mass loss as determined for all the experimental runs and their average values.

A quantitative analysis of the calcined *Ficus exasperata* leaves particles was carried out to determine the mineral content by a combination of XRF and EDX analysis. Table 3 presents the mineralogical properties and chemical constituents of the sample. XRF analysis showed that the silica purity of *Ficus exasperata* leaves ash was 67.50% while that of alumina was 6.50%. The ratio SiO_2/Al_2O_3 was 10:1. The results are in agreement and compare favorably with the results reported in Cordeiro et al. [16], who stated 78.340 and 8.550% for silica and alumina purity, respectively, resulting in SiO_2/Al_2O_3 ratio of 9:1. The results are also in conformity with some other previous researches in which were reported 87.40% SiO_2 and 3.60% Al_2O_3 [17]; 67.10%–76.80% SiO_2 and 3.43%–5.69% Al_2O_3 [8]; 78.34% SiO_2 and 8.55% Al_2O_3 [18]; and 70.5% SiO_2 and 5.23% Al_2O_3 [19].

3.1. X-ray diffraction and X-ray fluorescence analysis

XRD diffractometer was used to determine the zeolite patterns at

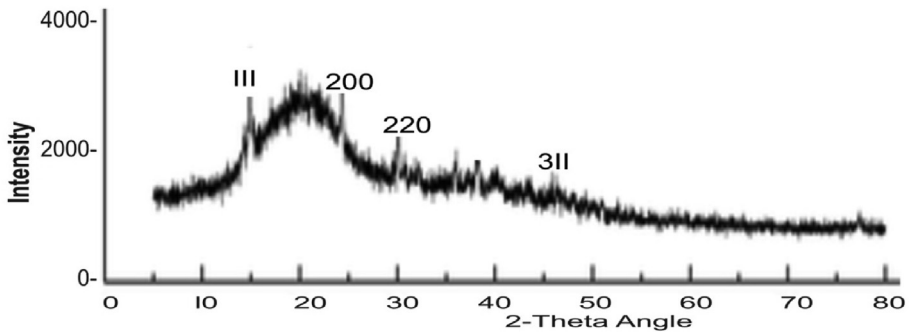

Fig. 1. XRD Pattern of *Ficus exasperate leaf ash.*

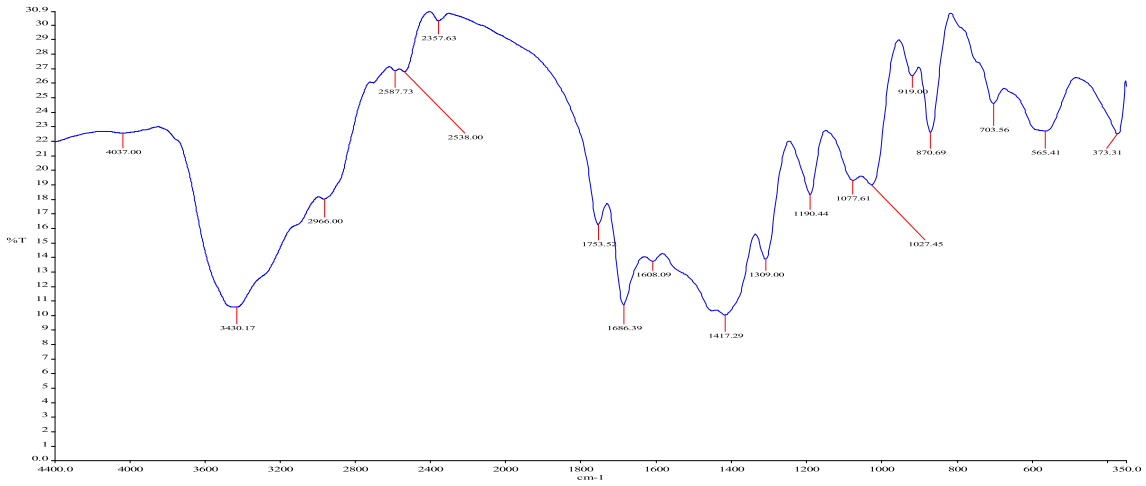

Fig. 2. FTIR spectra of synthesized Zeolite Y.

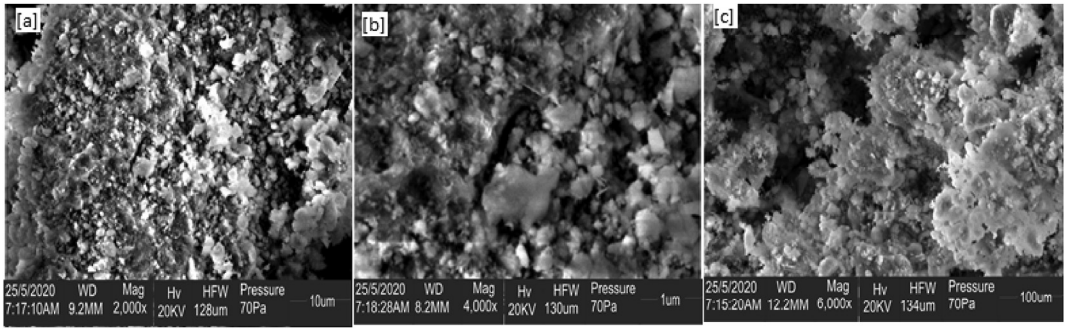

Fig. 3. SEM images of Synthesized zeolite-Y, (A) 4000x, (B) 2000x, (C) 6000x

standard of 2 theta range between 5 and 80° of 5 deg/min scanning speed and wave length λ = 1.5406 A° with Cu-Kα radiation source. The diffractograms of zeolite-Y formation phase was established by comparison with the diffractograms of the reference zeolite-Y [26]. Fig. 1 presents the XRD patterns of the calcined sample. Aluminosilicates of 74 wt.%, quartz (25 wt.%) and minor components (about 1 wt.%) were noticed in XRF data as presented in Table 3. The results indicate zeolite-Y constituent phase crystal structure as the major product obtained and intensities of XRD peaks pattern increased as given in the XRD data obtained [26].

3.2. FTIR analysis

Infrared technique was adopted to measure and determine the surface morphology and acidity of the sample. The region band between 4000

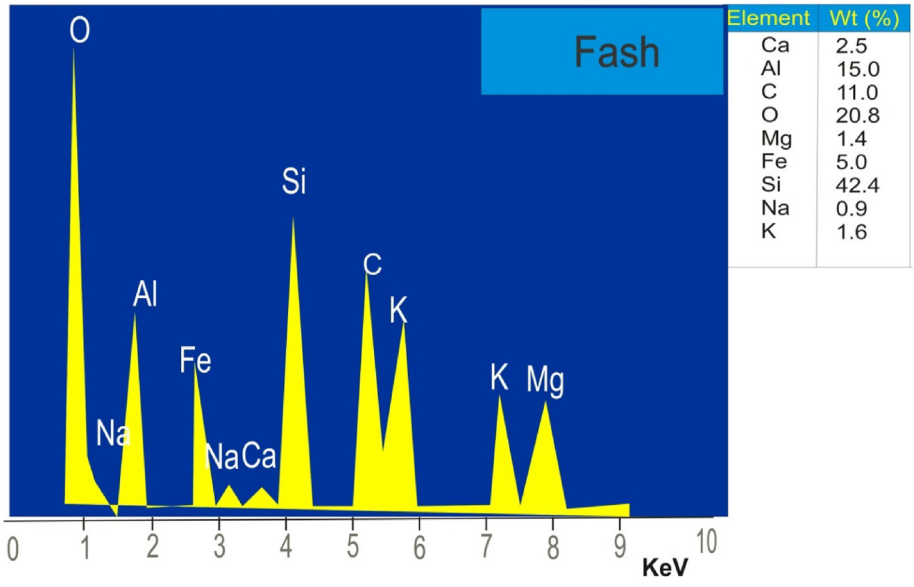

Fig. 4. EDX analysis of zeolite-Y.

and 3000 cm^{-1} reveals a medium sharp appearance of catalyst and OH stretching band, 3000–2840 cm^{-1} reveals C-H stretching band of the alkane functional group, the region between 3365 and 3489 cm^{-1} reveals OH stretching band and low frequency band of SiO_4 molecules with Al-OH. Aluminosilicates T–O bonds (T = Si or Al) was noticed between the shift band 1080 - 1000 cm^{-1} (1035, 1027,1004, 1003, 1001, 1000 and 996 cm^{-1}), which led to the conversion of SiO_2 and Al_2O_3 to aluminosilicates in the reaction of ash sample with NaOH [27]. The range band of 1010–1019 cm^{-1} reveals the presence of Si-O with 1600 cm^{-1} peak corresponding to water molecules bending vibration in zeolite structure and absorption in the range 443–465 cm^{-1} was allotted to Si–O – Al stretching. The result presented in Fig. 2 shows that FTIR spectrum of the synthesized zeolite ties with the typical absorption peaks of the commercial type [28]. Zeolitic materials are found and characterized between 1250 and 950 cm^{-1} asymmetric stretching vibration band. High crystalline zeolite and vibrations of functional groups of OH type were noticed within maximum bands of 3430 and 1608 cm^{-1} while frequencies near 1000 cm^{-1} are ascribed to asymmetric stretching of bonds Si–O or Al–O. Weak intensity broad band was noticed around 565.41 cm^{-1} to indicate zeolite Y cubic prism band presence and zeolite crystallization with double rings [29].

3.3. SEM/EDX analysis

SEM and EDX were used to determine the morphology and percentage weight of zeolite-Y, respectively. Micrographs were produced at specific magnifications of 2000x, 4000x, 6000x to show the surface morphology of the sample. Fig. 3a shows the morphology (mag. 2000x) of cubic crystalline micrographs, Fig. 3b (mag. 4000x) revealed granular crystalline microstructure surface and fine granular crystalline micrographs were observed in Fig. 3c with magnification 6000x, and are in good agreement with literature results [30]. The zeolitic products revealed, as shown in SEM micrographs and morphologies, possess a distinctive crystal pattern with established studies [29,30]. EDX analysis, as presented in Fig. 4, shows the cumulative percentage weight of significant aluminosilicate elements as 78.2% (Si = 42.4%, O = 20.8%, Al = 15.0%) while other elements gave a percentage weight of 21.8%, which confirms the presence of zeolite compounds [31–34].

4. Conclusion

It was affirmed that significant quantities SiO_2, and Al_2O_3 are embedded in *Ficus exasperata leaf ash*. The proportion of CaO and other trace functional constituents in minute quantities were also noticed, including P_2O_5, MgO and Fe_2O_3. Findings also showed that zeolite-Y type provides a crystalline formation with Na-Y functions. SEM/EDX analysis defined the morphology, uniform distribution, and high surface area (278.0250 m^2/g) compared with commercial zeolite surface area of 310.0906 m^2/g using BET analysis [28], and stable structure. Thus, based on the results of this study, *Ficus exasperata leaf* ash is a potential catalyst as it contains high silica and alumina contents with minimum of 67.50% and 6.50%, respectively, and could therefore be used for bio-oil cracking and stability improvement.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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