CONCLUSIONS

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The goal of this work was to use bagasse fly ash (BFA), a sugar industry waste that was obtained at little cost as a raw material for the synthesis of zeolitic composite adsorbents: CaFZBFA and MgFZBFA (by alkali fusion method); and CaMZBFA and MgMZBFA (by microwave alkaline hydrothermal method), with the incorporation of Ca and Mg ions (alkali earth elements), and investigate their adsorption potentials for the organic pollutants: aniline (AN); p-nitroaniline (PNAN) and nitrobenzene (NB), from aqueous solutions. The raw material, BFA used for the syntheses was found to contain very high silica content compared to the alumina content (SiO_2/Al_2O_3 = 54), hence sodium aluminate was used in the syntheses as aluminium supplement in order to improve the chances of zeolitization. The synthesized adsorbents were characterized by different instrumental methods and evaluated for their sorption performances. The outcomes of the experimental studies have led to the following conclusions:

- The FTIR analyses of the synthesized materials show the appearance of bands associated with zeolitic waters. In addition, the FTIR spectra established the polymerization interaction between silica and alumina constituent of BFA by the shift of TO_4 band present in the BFA spectrum at 1150 cm^{-1} to lower wavenumbers in the spectra of the synthesized materials, indicating the increase in the amount of tetrahedrally positioned Al in the synthesized materials. The manifestation of Si-OH group which is characteristic of zeolitic materials was also confirmed. These observations ascertain the successful transformation of the fly ash into zeolitic forms.

- XRF analyses show the increase in the moles of Ca in CaFZBFA and CaMZBFA; and the moles of Mg in MgFZBFA and MgMZBFA. This suggests the successful incorporation and Ca and Mg ions into the structures of the zeolites components of the adsorbents.
- The XRD diffractogram of BFA mainly shows peaks of amorphous and crystalline silica and mullite. In the XRD diffractogram of the synthesized adsorbents: CaFZBFA; MgFZBFA; CaMZBFA; and MgMZBFA, there is a disappearance of some of the silica and mullite peaks, most prominently the amorphous hump. There were appearances of new and crystalline peaks. These suggest that BFA had undergone some sort of transformations. Peaks of analcime, faujasite-Ca and sodalite were identified in the diffractogram of CaFZBFA. Peaks of faujasite-Mg and sodalite were detected in the diffractogram MgFZBFA. Peaks of sodalite and faujasite-Ca were detected in the diffractogram CaMZBFA. And peaks of chabazite-Mg and sodalite were identified in the diffractogram of MgMZBFA. Despite the confirmation of zeolitization of BFA, abundant silica peaks could still be observed in the diffractograms of CaMZBFA and MgMZBFA compared to CaFZBFA and MgFZBFA. This points out the lower efficiency of the dissolution of BFA particles for zeolitization processes in the microwave alkaline hydrothermal method.
- TGA results also confirm the zeolitic nature of the adsorbents, as the mass reduction steps during analysis: the loss of adsorbed water from the outer pores; the loss of water of crystallization associated with zeolitic cations; and the loss of water due to thermal dehydroxylation process, are peculiar to zeolites. In addition, the basis for insight into the degree of conversion from fly ash to zeolite phases is the mass loss of the adsorbents during thermal treatments. The desorbed water

content reflects the free volume in the zeolite cavities which could be accessible to adsorbates which could be used as a measure of the degree of conversion of fly ash to zeolites. The adsorbed/desorbed waters quantified in the fused products are more than those quantified in the microwave alkaline hydrothermal products. Hence, it can be said that the degree of zeolitization in the adsorbents whose synthesis involve alkali fusion is more than that of the adsorbents synthesized by microwave alkaline hydrothermal method.

- Results of the physicochemical properties of the adsorbents show that CaFZBFA and MgFZBFA have better surface areas (124 m^2/g and 83 m^2/g respectively) than CaMZBFA and MgMZBFA (4 m^2/g and 3.26 m^2/g respectively). Likewise, the pore volumes of the former adsorbents are better than those of the latter. Hence, it could be said that the methods of converting BFA to zeolitic forms have great influence on some properties such as the surface area, porosity and bulk density that could have a significant effect on adsorption performances. Furthermore, sorption experiment and isotherms results reveal that CaFZBFA have the best set of maximum adsorption capacities Q_L for the pollutants (34.13 mg/g, 32.36 mg/g and 21.60 mg/g for AN, PNAN and NB respectively), followed by MgMZBFA (33.22 mg/g, 30.86 mg/g and 19.92 mg/g for AN, PNAN and NB respectively), followed by CaMZBFA (16.08 mg/g, 15.92 mg/g and 12.95 mg/g for AN, PNAN and NB respectively) and the least MgMZBFA (14.99 mg/g, 12.72 mg/g and 10.20 mg/g for AN, PNAN and NB respectively).
- pH also seemed to be influential towards the adsorption of the pollutants from aqueous solutions by CaFZBFA, MgFZBFA, CaMZBFA and MgMZBFA. Sorption studies showed that the optimum sorption of AN (27.0 mg/g, 24.9 mg/g, 14.8 mg/g and 12.5 mg/g for

CaFZBFA, MgFZBFA, CaMZBFA and MgMZBFA) was at pH 6. Optimum sorption of PNAN (24.1 mg/g, 21.0 mg/g, 12.2 mg/g and 9.5 mg/g for CaFZBFA, MgFZBFA, CaMZBFA and MgMZBFA) was at pH 4. And the optimum sorption of NB (21.0 mg/g, 19.2 mg/g, 11.1 mg/g and 8.1 mg/g for CaFZBFA, MgFZBFA, CaMZBFA and MgMZBFA) was obtained at pH 9. These results suggest that the nature of the pollutants has an effect on their adsorption performance. AN which is the most electronegative among the other pollutants gave the best adsorption performances and seemed to interact best with the positive surfaces of the adsorbents (at pH < PZC) while NB was least adsorbed by all the adsorbents.

- Intraparticle diffusion plots for the sorption processes involving the pollutants: AN; PNAN; and NB, and the adsorbents: CaFZBFA; MgFZBFA; CaMZBFA; and MgMZBFA all show dual distinct linear sections. This means that diffusion of the pollutants through the pores of the adsorbents complements surface adsorption in the adsorption mechanism of each of the adsorption systems.
- Temperature has an influence on the adsorption processes. The adsorption capacities q_e of CaFZBFA, MgFZBFA, CaMZBFA and MgMZBFA increased with increase in temperature (298 – 328K) for the adsorption of AN and PNAN. Also, the calculated ΔH^o values for the adsorption processes are positive. Hence, the adsorptions of AN and PNAN on CaFZBFA, MgFZBFA, CaMZBFA and MgMZBFA are endothermic. The adsorption capacities q_e of CaFZBFA, MgFZBFA, CaMZBFA and MgMZBFA reduced with an increase in temperature for the adsorption of NB. And the calculated ΔH^o values for the adsorption processes are negative. Therefore, the adsorption of NB on

CaFZBFA, MgFZBFA, CaMZBFA and MgMZBFA are exothermic in nature.

- The regeneration studies carried out on the adsorbents showed that they could be reused multiple times. This with the facts that: the adsorbents exhibit better performances (Q_L) than some previously studied adsorbents, and were synthesized from raw materials which are wastes and obtained at little cost, makes the application of the adsorbents a potential large-scale economic means of sequestrating the pollutants: aniline; p-nitroaniline; and nitrobenzene from contaminated waters.