

## CHARACTERIZATION OF ZINC CHLORIDE MODIFIED ACTIVATED CARBON AND OTHER DERIVATIVE ADSORBENTS SYNTHESIZED FROM *VITEX DONIANA* SEED

\*Francis, A. O.<sup>1</sup> and Otoikhian, S. K<sup>2</sup>

<sup>1,2</sup>Department of Chemical Engineering, Edo State University, Uzairue, Nigeria

\*Email: francis.asokogene@edouniversity.edu.ng

### ABSTRACT

*This study evaluated the characteristics and adsorption performance of zinc chloride modified activated carbon (VDZnCl<sub>2</sub>), its precursor (VDC), and sodium hydroxide (VDNaOH), hydrochloric acid (VDHCl) and thermally (VDT) modified adsorbents from vitex doniana. The adsorption performance of the adsorbents for methylene blue was in this order: VDZnCl<sub>2</sub> < VDNaOH < VDC < VDT < VDHCl. The VDZnCl<sub>2</sub> was characterized by Fourier transform infrared (FTIR), Brunauer-Emmett-Teller (BET), scanning electron microscope (SEM), energy-dispersive X-ray (EDAX) and thermal gravimetric analysis (TGA). The FTIR spectrum showed the presence of O—H group and characteristics C=C group generally found in carbonaceous materials. The BET surface area remarkably increased from 14.02 m<sup>2</sup>/g to 933.25 m<sup>2</sup>/g and pore size from 0.92 to 18.9 Å which reflected enhanced specific surface area and porous nature of the adsorbent, and its ability to facilitate pore fillings of many molecules inside its carbon matrix during adsorption. The SEM micrograph showed varieties of pores with widened cavities. Therefore, VDZnCl<sub>2</sub> is a potential adsorbent substitute for wastewater treatment.*

**Key words:** *Vitex doniana, characteristics, activated carbon, adsorption, carbonaceous materials*

### 1. INTRODUCTION

The growing trend of water contaminations resulting from growing population and industrialization are of great concern because they contaminate surface and ground water, and also render them unfit for drinking and irrigation when they are discharged into streams, oceans and lakes by chemical, textile and other allied industries (Piriya et al. 2021). These contaminants such as heavy metals, dyes etc. in water limit sunlight penetration into water and aeration of water body which impact negatively on photosynthesis and subsequently reduce the amount of dissolved oxygen in water (Piriya et al. 2021; Imran et al 2019). They also inject chemicals which leads to the death of most aquatic life, contamination of soils, poisoning of drinking water which results in numerous health issues like dyscrasia, leukocytosis, anemia, eye burn, damage to the liver, heart, spleen, kidney, lungs, bones and teratogenic effects (Alipour et al. 2019; Piriya et al. 2021).

Several treatment methods adopted for the removal of contaminants from wastewater include extraction, membrane separation, coagulation, ozonation, flocculation and adsorption (Asokogene et al. 2019; Aslam et al. 2017; Luo et al. 2017; Wang et al. 2017). Adsorption holds special place in the treatment of wastewater because of its low cost, effectiveness and ability to selectively enrich certain compounds

(Asokogene et al. 2019; Pandey et al. 2020; Piriya et al. 2021). This has led researchers to develop low cost and readily available adsorbents from several synthetic and natural biomass.

Activated carbon has gained wide attention as brilliant adsorbent for the removal of aquatic pollutants due to its high surface area, stability, array of functional groups and excellent pore structures (Afshin et al. 2019). It is produced from several agricultural wastes such as empty fruit bunch (Zaini et al., 2016), woods (Nowicki, 2016) bamboo (Zhao et al. 2017), palm shells (Zhao et al. 2018), coconut shell (Singh et al. 2017), rich husk (Li et al. 2015), sawdust (Zhu et al. 2014), apricot stones (Djilani et al. 2015) and grape seeds (Okman et al. 2014) etc. by chemical or physical activation. Chemical activation enhances its specific surface area and porosity (Piriya et al. 2021). Zinc chloride has proven to be a better activating agent for most activated carbon adsorbent as it produces higher specific surface area and better adsorption results. For instance, ZnCl<sub>2</sub> activated carbon from sterculia gulata shells had adsorption capacity of 90.90 mg/g as against the precursor (sterculia gulata shells) of 45.45 mg/g (Rangabhashiyam and Selvaraju, 2015). Similarly, Zhang et al. 2020 also reported that ZnCl<sub>2</sub> impregnated activated carbon from rice husk at impregnation ratio of 1:1 had higher surface area than its precursor.

Consequently, *vitex doniana* seed, which are frequent waste in Nigeria (Ameh et al. 2012; Kapooria and Aime, 2005) and obtained from commonly grown *vitex doniana* tree has been limitedly synthesized as activated carbon adsorbent for Zn (II) and Pb (II) removal (Ameh et al. 2012) and as acid modified activated carbon for Cr (II) removal (Yusuf et al., 2020) from wastewater, but no study has been done on the synthesis of ZnCl<sub>2</sub> modified activated carbon from *vitex doniana* seed and its adsorption potential for dye (methylene blue) from wastewater. Therefore, this study was a first-time attempt of the synthesis of zinc chloride (ZnCl<sub>2</sub>) modified activated carbon adsorbent from *vitex doniana*, removal of methylene blue dye from wastewater and comparing its adsorption performance its precursor, sodium hydroxide (NaOH), hydrochloric acid (HCl) and thermally modified ones, with the view to encourage its use as indigenous adsorbent for wastewater treatment, thus contributing to sustainable development goals. BET, SEM, EDX, FTIR and TGA were used to determine the properties of the adsorbent and its adsorption potential in batch mode was carried out on methylene blue removal from wastewater within a working concentration of 10 mg/L.

## 2. MATERIALS AND METHODS

### 2.1 Materials, reagent and instrument

*Vitex doniana* seeds were collected from Auchi Polytechnic community in Edo State. The chemicals used were of analytical grade: hydrochloric acid (37 %, Fisher Scientific, USA), methylene blue powder (98.5 %, BDH, England), Zinc chloride pellet (99%, Merck, Germany) and distilled water (Chemical Engineering laboratory, Auchi). Instruments used are analytical balance (Scout Pro, Ohaus, London, UK), pH meter (pH ep® pocket-sized pH meter, Hanna Instruments, Inc., USA), grinding mill (Biocotek, China), shaker (Ro-tap, England), stop watch (Quartz, China), Thermometer (Pyrex Technico, England), Fourier transform infrared (FTIR) spectrometer (Thermo Scientific, Nicolet ISI 10, USA), scanning electron microscope (SEM)/ energy dispersive analysis of x-ray (EDX) spectrophotometer (Karl Zeiss, Germany), Brunauer-Emmett-Teller (BET) surfer machine (Thermo Scientific, USA), thermogravimetric analyzer (Orton Simultaneous DTG/TGA, USA), UV-Vis spectrophotometer (Angstrom Advanced Inc, model 752, Massachusetts, USA) and muffle furnace (TT-EF-12, Techmel, USA).

### 2.2 Pre-treatment and synthesis of *vitex doniana* adsorbent

The exocarp and impurities of *vitex doniana* seed were washed off with running water. The seed was sun-dried for seven days, crushed, ground, and sieved to a size of 355 µm (Ameh et al. 2012). A 100 g each of the pulverized *vitex doniana* was treated thermally at 800 °C (Mistar et al. 2020), and chemically modified using ZnCl<sub>2</sub>, sodium hydroxide (NaOH) and hydrochloric acid (HCl) by adding 0.5 M solution of each chemical to the pulverized samples in the ratio of 1:1 to form homogenize paste-like mixtures which were allowed to stay for 24 h for adequate impregnation. The mixtures were activated at 800 °C for 2 h in a TT-EF-12 muffle furnace (Techmel, USA). The resultant activated carbon samples were allowed to cool, washed with distilled water to a pH of 7, dried in the oven at 105°C for 3 h and kept in a desiccator, except for ZnCl<sub>2</sub> modified sample which was soaked in HCl (3 wt%) for 12 h to remove surface ash before washing (Mkungunugwa et al. 2021; Mistar et al. 2020; Jaria et al. 2015; Khadiran et al., 2015). These adsorbents were designated as VDC, VDT, VDZnCl<sub>2</sub>, VDNAOH and VDHCl for precursor, thermally treated, ZnCl<sub>2</sub> modified, NaOH modified and HCl modified *vitex doniana*, respectively.

### 2.3 Characterization of *vitex doniana* adsorbent

The surface functional groups of VDZnCl<sub>2</sub> and VDC (precursor) adsorbent samples were determined using FTIR analyzer (Thermo Scientific Nicolet ISI 10, USA) at wavenumber range of 4000-500 cm<sup>-1</sup>. The textural properties were measured using a Thermo Scientific (USA) surface area analyzer. The SEM image of the adsorbent surface and texture was obtained by a Karl Zeiss (Germany) instrument. The thermal decomposition of the adsorbent sample was determined using the standard Orton simultaneous DTG/TGA analyzer, USA by recording the differential thermal couple output and balance output signal over temperature range of 30–1000°C. The elemental composition of the adsorbent was also obtained using EDX as individual fluorescent energies detected were specific to the elements that were present in the sample.

### 2.4 Experimental procedure

For batch adsorption study, 1 g of methylene blue powder was dissolved in 1000 mL of distilled water to make stock solution and working concentration of 10 mg/L was prepared by dilution. In Beatson bottles, 50 mL of the concentration was added to 50 mg of the adsorbents. Next, the bottles were sealed, shaken, and

kept at 28°C for 72 h. The contact time was assumed to be long enough to achieve equilibrium adsorption (Asokogene et al. 2019). The residual concentrations were calculated by measuring absorbance at 620 nm with an Angstrom Advanced Inc. (model 752) scientific ultra violet-visible (UV-Vis) spectrophotometer. The pH of the methylene blue solution was kept at 4.8±0.3, as it should be. The adsorption capacity,  $q_e$  (mg/g), was determined from a mass balance equation,

$$q_e = \left( \frac{C_o - C_e}{m} \right) \times V \quad (1)$$

where  $C_o$  and  $C_e$  (mg/L) are the initial and equilibrium concentrations, respectively,  $V$  (L) is the volume of solution, and  $m$  (g) is the mass of the adsorbent.

### 3. RESULTS AND DISCUSSION

The batch adsorption plots to determine the best among the series of prepared adsorbents from *vitex doniana* (VDNaOH, VDZnCl<sub>2</sub>, VDHCl, VDT and VDC) is presented in Figure 1. The result revealed that VDNaOH, VDZnCl<sub>2</sub>, VDHCl, VDT and VDC adsorbents had adsorption capacities of 8.57 mg/g, 9.08 mg/g, 5.01 mg/g, 5.40 mg/g and 7.98 mg/g, respectively. In other words, the adsorption capacities were of this order: VDZnCl<sub>2</sub> < VDNaOH < VDC < VDT < VDHCl. This better adsorption performance demonstrated by VDZnCl<sub>2</sub> can be attributed to the creation of better active reaction sites for adsorption of methylene blue in VDZnCl<sub>2</sub> than VDNaOH, VDC, VDT and VDHCl (Ademiluyi and David-West, 2012).

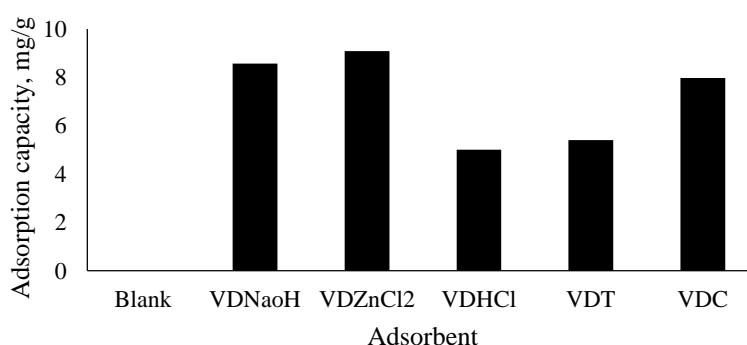


Figure 1: *Vitex doniana* adsorbents performance

The FTIR spectrum which gave insight into the surface functional groups and structure of VDZnCl<sub>2</sub> and VDC adsorbents over frequency range of 4000-500 cm<sup>-1</sup> are presented in Figures 2 and 3. The signal at 3625–3605 cm<sup>-1</sup> in the FTIR spectrum corresponds to intermolecular hydrogen bonding of polymeric compounds such as alcohols, phenols, and carboxylic acids, showing the presence of free hydroxyl groups on the adsorbent surface of VDZnCl<sub>2</sub> and VDC. Asymmetric and symmetric C—H bending vibrations of the —CH<sub>2</sub> and —CH<sub>3</sub> (alkanes) are associated with a peak at 3300 cm<sup>-1</sup> of VDZnCl<sub>2</sub> while the peak at 2410.16 cm<sup>-1</sup> corresponds to the CH<sub>2</sub> (alkanes) group of C—H symmetric stretching vibration for VDC. Furthermore, the C=C stretching vibration of alkenyl is the characteristic of a peak at 2611 cm<sup>-1</sup> for VDZnCl<sub>2</sub>. Meanwhile, C=O stretching vibration in aldehydes, ester or ketones, and carboxyl groups are indicated by the peaks at 1877 cm<sup>-1</sup> and 1903.27 cm<sup>-1</sup>, respectively of VDZnCl<sub>2</sub> and VDC.

Because of the carboxyl group, the peak at 1710.75 cm<sup>-1</sup> corresponds to C—O stretching vibrations, while the C—O stretching vibrations in alcohol, ether, or hydroxyl groups correlate to the peak at 1150.28 cm<sup>-1</sup> for VDC. The peak at 1551 cm<sup>-1</sup> of VDZnCl<sub>2</sub> corresponds to C=C

stretching vibrations in the aromatic ring of alkenes, which are common in carbonaceous materials like activated carbon (Mkungunugwa et al. 2021). Finally, the peak at 1298 cm<sup>-1</sup> of VDZnCl<sub>2</sub> belongs to C—H bending vibrations found in alkyl groups like CH<sub>3</sub>, which are lignin-like.

The functional groups contained in VDZnCl<sub>2</sub> and VDC revealed that majority of the peaks in both adsorbent materials were comparable, with the exception of a peak at 1551.37 cm<sup>-1</sup>, which corresponds to C=C stretching vibrations in the aromatic ring of alkenes and is commonly detected in carbonaceous materials like activated carbon (Mkungunugwa et al. 2021).

Characterization Of Zinc Chloride Modified Activated Carbon And Other Derivative Adsorbents Synthesized From *Vitex Doniana* Seed

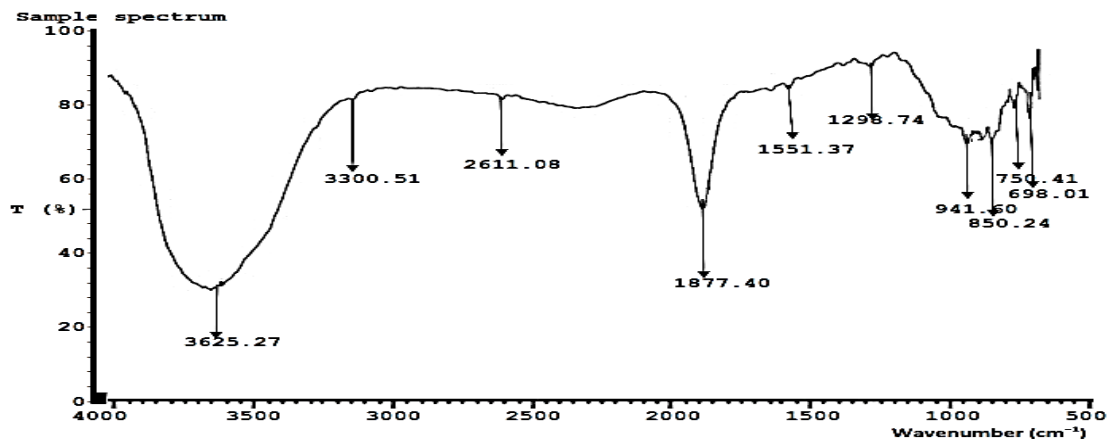


Figure 2: FTIR spectrum of zinc chloride modified vitex doniana activated carbon (VDZnCl<sub>2</sub>)

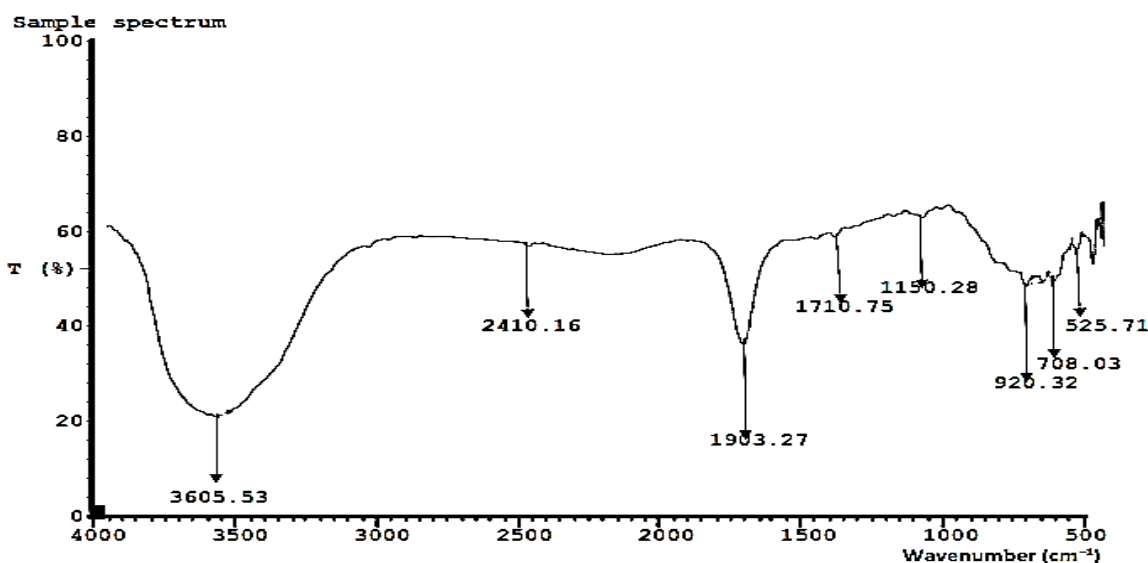


Figure 3: FTIR spectrum of vitex doniana precursor (VDC)

Table 1 summarizes the textural properties of VDZnCl<sub>2</sub> and VDC. The surface area of VDZnCl<sub>2</sub> increased dramatically from 14.0 to 933 m<sup>2</sup>/g. The pattern is consistent with other raw materials upon activation (Mistar et al. 2020; Gonzalez, 2018). Accordingly, the pore volume increased by about three times, from 0.07

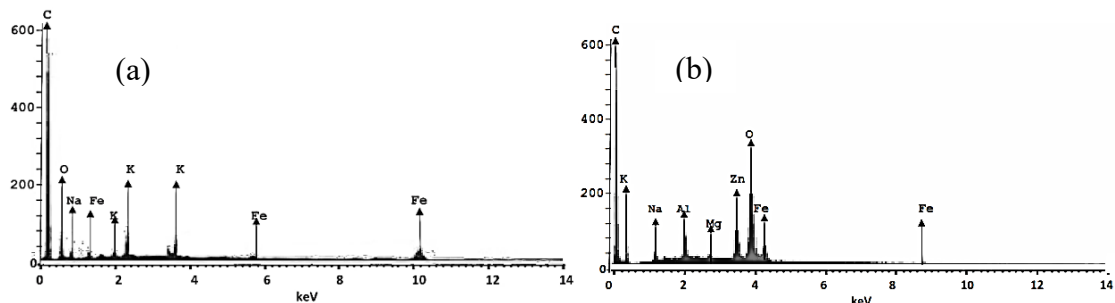
to 0.21 cm<sup>3</sup>/g (Mistar et al. 2020; Sethia and Sayari, 2016). After activation, the pore size increased from 0.92 to 18.9 Å. Therefore, VDZnCl<sub>2</sub> will allow for pore fillings of numerous molecules inside its carbon matrix during adsorption ((Thommes et al. 2015; Mistar et al. 2020).

Table 1: Surface Textural Properties of VDZnCl<sub>2</sub> and VDC

BET Parameters	Samples	
	VDC	VDZnCl <sub>2</sub>
Surface area (m <sup>2</sup> g <sup>-1</sup> )	14.02	933.25
Pore volume (cm <sup>3</sup> g <sup>-1</sup> )	0.0683	0.207
Pore size (Å)	0.917	18.92

The SEM result of VDZnCl<sub>2</sub> adsorbent material which was characteristics of its surface morphology and texture at ×500 magnification is shown in Figure 4 and the EDX

results of VDZnCl<sub>2</sub> and VDC are shown in Figures 5 (a) and (b), respectively.

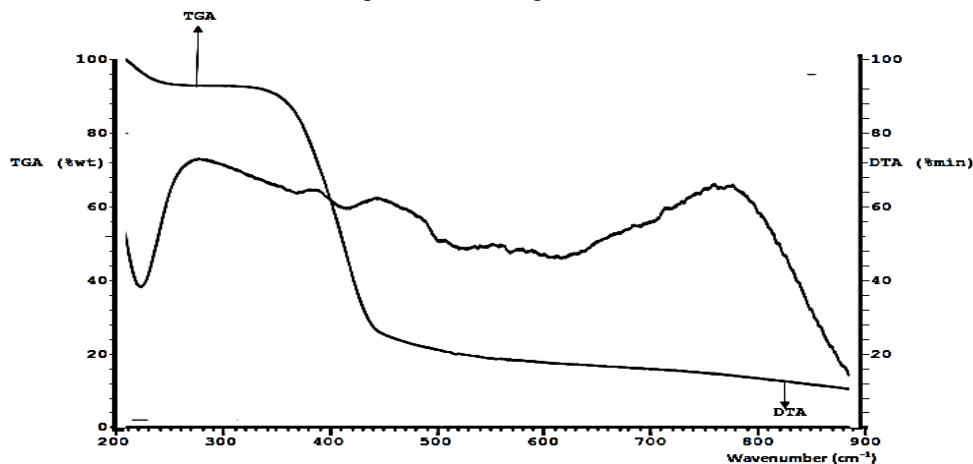
Figure 4: SEM micrograph of VDZnCl<sub>2</sub> adsorbentFigures 5: EDX analysis of (a) VDZnCl<sub>2</sub> adsorbent and (b) VDC adsorbent

The SEM micrograph of VDZnCl<sub>2</sub> adsorbent showed thin plant boundary walls due to modification and the presence of varieties of pores with widened cavities which could be due to the removal of volatile matter and production of fixed carbon after activation (Ohimor et al. 2021).

The EDX result revealed dominance of C and O in the precursor (VDC) and activated carbon adsorbent (VDZnCl<sub>2</sub>). However, in VDC small proportion of Mg, Al, Ca and Zn were observed which were not present in

VDZnCl<sub>2</sub>. These elements would have been removed along with other volatile materials during activation at 800°C. Alam et al. 2017 reported similar result for activated carbon synthesized from *Eucalyptus lenceolata*.

The TGA/DTA result for ZnCl<sub>2</sub> based *vitex doniana* activated carbon, which was characteristics of its thermal behavior at regulated temperature is shown in Figure 6.

Figure 6: Thermal gravimetric profile of VDZnCl<sub>2</sub>

The curves showed three steps weight loss (Figure 6) from 200 to 900°C and 48.90 to 68%. The first weight loss within temperature range of 200-250°C could be due to the eradication of water vapour and light volatile matter (Piriya et al. 2021). The second weight loss within temperature range of 350 to 500°C could be as a result of liberation of organic constituents and other functional groups in the adsorbent. Finally, the less pronounced third weight loss within temperature range of 650 to 700°C was probably due to heavy volatile matter eradication or disintegration of high strength configuration (Piriya et al. 2021). Nevertheless, decomposition of the adsorbent material occurred slowly from 200 to 900°C with most of its fraction been lignin and residual traces of possibly cellulose and hemicellulose (Piriya et al. 2021; Cazetta et al. 2011).

#### 4. CONCLUSION

Zinc chloride modified activated carbon adsorbent (VDZnCl<sub>2</sub>) and other derivative adsorbents (VDC, VDT, VDNaOH and VDHCl) were synthesized from *vitex doniana* seeds which are common waste around Auchi Polytechnic community in Edo State. The adsorption performance of the adsorbents for methylene blue was in this order: VDZnCl<sub>2</sub> < VDNaOH < VDC < VDT < VDHCl. Therefore, VDZnCl<sub>2</sub> was characterized for its physicochemical properties. The presence of O—H group, characteristics C=C group generally found in carbonaceous materials, improved surface area and pores with widened cavities reflected its potential as adsorbent for methylene blue. The textural properties showed tremendous increase after modification from 14.0 to 933 m<sup>2</sup>/g, the pore volume increased by about three times, from 0.07 to 0.21 cm<sup>3</sup>/g and the pore size increased from 0.92 to 18.9 Å.

#### 5. ACKNOWLEDGEMENT

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**Characterization Of Zinc Chloride Modified Activated Carbon And Other Derivative Adsorbents Synthesized From *Vitex Doniana* Seed**

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