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*Keywords: physicochemical activation; porosity; functional group; low-cost adsorbent; activated carbon.*

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*Abstract-* Low-cost adsorbents (from Oil Palm Shell (OPS) and polyethyleneterephtalate (PET)) were prepared by physical activation with steam and Physico-chemical activation using both KOH and steam in the way to study the modification of porosity, pores size distribution and surface functional group. The activated samples obtained by physical activation are microporous with a surface area between 1000and 1500 m2/g, while the other obtained by physicochemical activation contain a well-developedporosity which consists of micropores, mesopores and likely macropores with a surface area between 991 and 1413 m2/g. The adsorption of methylene blue (MB) and iodine (I2) show that physicochemical activation produced a strongly developed internal surface, which is open at the surface by, mesopores and possibly macropores. FTIR spectrum of ACs prepared shows many peaks belonging to different functional groups. Samples obtained by physicochemical activation give more intense peaks than those obtained by physical activation. It was found that the use of KOH permits incorporation of O-containing groups at the surface of carbon material. During Raman and DRX analysis, it appears that physicochemical activation creates more defects in the structure of carbon materials than those obtained by physical activation method; carbon atoms are eliminated and allow connecting internal pores to others and the external pores at the surface. These results show that the use of KOH during physical activation significantly develops the porosity and functional group at the surface of the activated carbons obtained.

*Keywords: physicochemical activation; porosity; functional group; low-cost adsorbent; activated carbon.*

## I. INTRODUCTION

The increasing demand for porous materials for environmental remediation has focused the efforts to the development of new methods of preparation of activated carbon. Activated carbons are widely used adsorbents; this is attributed to their well-developed internal porosity, large surface area and multiples functions at the surface which are active sites [1, 2]. Activated carbons can be produced from any carbon-containing organic materials, but the structure of the obtained carbon is closely linked to the structure of the raw material. However, the best raw materials for the preparation of the good activated carbon are materials having a high content of carbon and inexpensive [3,4]. As a result, wastes of lignocellulosic origin [5-9] and plastics wastes [10-14] are used, to obtain the low-cost active carbon.

The porous texture and functional groups on the surface of the AC also depend on the preparation process. There are many studies on the preparation of AC which are grouped in two: (i) Chemical activation, which consists of impregnation of the raw material with a strong dehydrating agent (KOH, $H_3PO_4$, ZnCl ...) before carbonization. (ii)The physical activation, in this process the precursor is first carbonized at a temperature between 500 to 700°C under inert atmosphere ($N_2$ gas) and then activated in a stream of steam or carbon dioxide or air [15-17]. Whatever the method used is varied some parameters such as the final heating temperature, the temperature rise rate, the residence time at the final temperature, the thermal conductivity of the precursor [2].

Research on the preparation of activated carbons frequently uses the two methods mentioned above, by varying the precursor and/or the preparation parameters. It is therefore important to explore new preparation methods by combining these two classical methods of preparation and to study its impact on the quality of the adsorbent material obtained.The economic advantage of the use of activated carbons in water treatments based on adsorption processes can be enhanced by exploring new synthetic routes or using nonvaluable by-products, residues or post-consumer waste materials as precursors for their preparation [10].

In the present investigation, different types of activated carbon were prepared using lignocellulosic waste and post-consumer plastics. The objective is to increase the porosity of the obtained adsorbent during

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the preparation process. Two methods of preparation will be used, physical activation with water vapour, which is a very well-known method for generating micropores into AC [1-5], on the other hand a new method will be experimented by combining the method of physical activation with the method of chemical activation that we designate in this work by physicochemical activation method.

## II. EXPERIMENTAL METHODS

### *a) Preparation of activated carbon*

The precursors of ACs prepared are both Post-consumer plastic in polyethyleneterephtalate(PET) and Oil Palm Shell (OPS). The OPS samples grinned into small particle sizes ranging between 0.5 to 1.5 mm in diameter, dried at 110°C for 24hrs in the oven to remove all the moisture content weight and PET samples were cut down to desirable particle sizes and dried in sunlight for 5hrs. OPS and PET were mixed in the weight ratios 1:1 and 2:1 and were then subjected to carbonization using a cylindrical furnace. The heating started at room temperature with a temperature increment rate of 10°C/min up to a final temperature of 600 °C. The heating process was stabilized with nitrogen at a flow rate of about 100 mL/min. The residence time of the samples at the final stabilized temperature of 600°C was 3hrs. After heat treatment, the nitrogen flow was stopped, and the charcoals were cooled down to room temperature. Four carbonized compounds were obtained; one from OPS, one from PET, two from the mixture of the two in the ratio 1/1 and ½.

#### i. *Physical activation*

In the activation process, 5–6 g of the char were suggested to physical activation with steam (0,13 mL/min) in a furnace by heating the reactor from room temperature to 850 °C at a heating rate of 10 °C/min, with a residence time of 2 hrs.

#### ii. *Physico-chemical activation*

Before the physical activation step, 5–6g of the carbonized samples were mixed with Potassium hydroxyl in the ratio 1:2 (char/KOH) and dried in the oven overnight at 110ºC. The mixtures were activated with steam (0,13mL/min) in a furnace by heating the reactor from room temperature to 850ºC at a heating rate of 10ºC/min, with a residence time of 2hrs. After the activation, the steam flow was stopped, and the obtained ACs were cooled down to room temperature and washed with distilled water to a pH of 7.

### *b) Characterization of Activated Carbon*

Textural characterization was performed by adsorption–desorption $N_2$ isotherms at – 195, 8 °C. The adsorption isotherms experiments were performed by MICROMERITICS TRISTAR 3000. Corresponding surface area $S_{total}$ and Sext, micropore area $S_{micro}$ and volume $v_{micro}$, total pore volume and pore diameters were obtained by Brunauer–Emmet–Teller (BET), Harkins–Jura (HJ), t-plot method and *Barrett-Joyner-Halenda (BJH)* methods, respectively. For BET analysis, experimental points measured at a relative pressure of nitrogen gas $p/p^0$ below 0.3 on the adsorption branch were used. Fourier-transform infrared spectroscopy (FTIR), Raman spectra, transmission of electron microscopy (TEM), and X-ray diffraction (XRD) were also performed to characterize the samples.

### *c) Adsorptions tests*

The iodine number ($I_2$) of activated carbon was obtained by the Standard Test Method by titration with sodium thiosulphate. By batch experiment, methylene blue (MB) solution was mixed with activated carbon and shaken at the rate of 200 rpm at room temperature. After the completed reaction, the solutions were filtered, and the residual concentrations of methylene blue solutions were then determined by using a spectrophotometer at 660 nm. The equilibrium adsorption capacities ($Qe$) of the AC were determined based on the adsorbate mass balance using Equation 2:

$$Qe = \frac{(Co - Ce)V}{m} \qquad (1)$$

Where *Co* and *Ce* are the initial and equilibrium concentrations of the dye (mg/L) respectively, *V* is the volume of the aqueous solution (L), and *m* (g) is the mass of activated carbon used.

## III. RESULTS AND DISCUSSION

Eight samples of activated carbon were obtained in this work: four samples prepared by physical activation from lignocellulosic material and post-consumer plastic named OPSAC and PETAC and from their mixture the ratios 1:1 and 2:1 respectively named COM1 and COM2; equally four sample with the same precursor prepared by physicochemical activation named OSPIAC, PETIAC, COM1I, and COM2I.

### *a) Adsorption-desorption isotherms*

Figure 1 and figure 2 show respectively the adsorption isotherms of nitrogen obtained from AC prepared from the pure raw material and their mixture.

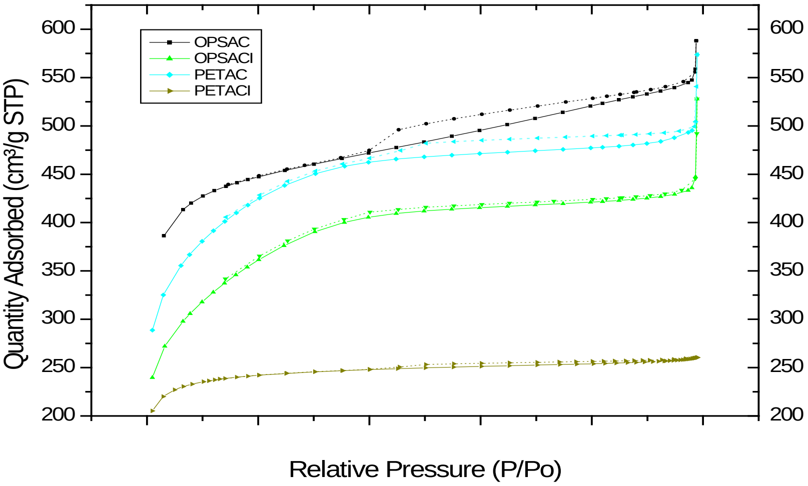


*Figure 1:* $N_2$ adsorption–desorption isotherms of ACs obtained from oil palm shell and PET by both physical activation and physicochemical activation method with steam and KOH

Adsorption-desorption of nitrogen is an important tool to analyze the structure of the prepared carbon. The adsorption in the gas phase shows clearly that the two samples prepared by physical activation adsorb better than those obtained by physicochemical activation (Figure1). Indeed the adsorption isotherms of the samples OPSAC and PETAC indicate the higher nitrogen volume adsorbed at low relative pressure. The knee shape at relative pressures close to unity shows a more rapid increase in adsorption due to the phenomenon of capillary condensation in mesopores. This can be explained by the formation of hysteresis (Figure.1), the pronounced intensity of hysteresis imply the presence of mesopores or macropores at the surface. On the other hand for the two samples obtained by physicochemical activation from the same raw materials, they adsorb less than the others, and the hysteresis is almost not visible. This shows that these two samples are essentially microporous, the mesopores if they exist serve as the gateway to the micropores, this shows the existence of a highly developed network of porosity[18-20].

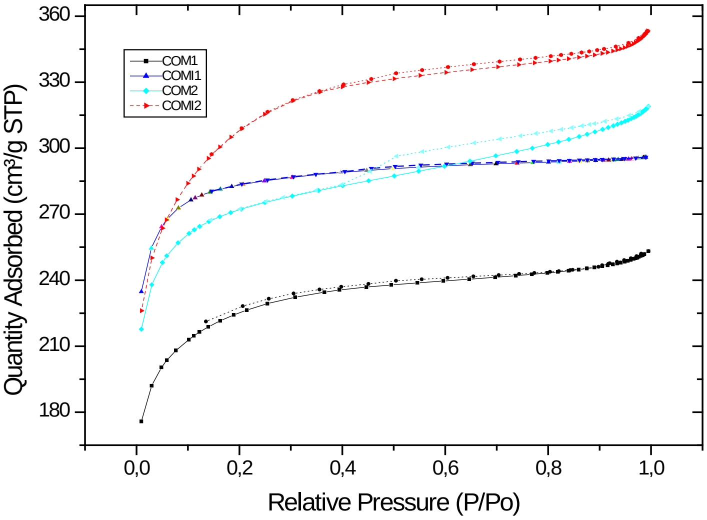


*Figure 2:* $N_2$ adsorption–desorption isotherms of ACs obtained from the mixture of oil palm shell and PET in the ration 1/1 and ½ by both physical activation and physicochemical activation method with steam

The composite samples behave differently from those prepared from pure raw materials; figure 2 shows that the samples obtained by physicochemical activation adsorb better than those prepared by physical activation. It is difficult to explain this phenomenon. However, we can assume that during the carbonization process, gaseous and volatiles compounds evolved from the solid, followed by a restructuring of the carbonaceous structure between the two constituents of the mixture, during this process, the formation of soot can appear. The char is constituted of a group of disordered short graphitic crystals containing micropores, which are often not accessible from the external surface because the mesopore network is blocked by soot deposition.The impregnation of the char bye KOH before the activation allow to remove soot and free carbon at the external surface and render micropore accessible. As we have seen for the carbons prepared from the pure precursors, the physicochemical activation generates materials possessing essentially the micropores.

Surface area and porosity values of the activated carbons are presented in Table 2. It shows that the activated carbons had remarkable BET surface area, due to the high micropore content [18-20]. Porosimetry parameters such as micropore area, micropore volume, cumulate volume and pore diameter were also compiled in Tables 2. It can be seen that the ACs had remarkable BET surface area in agreement with micropores content. The micropore area contribution is around 70% for AC prepared by physical activation, these quantities of micropores reduce (25.59 for OPSACI and (36.6) for PETACI) when the same raw material is submitted to the physicochemical activation. This can be explaining the fact that the activation process using both steam and KOH enhances the volume and enlarges the diameters of the pores. We can also suggest that impregnation with KOH widen existing micropores and the formation of large pores by burnout of the walls between the adjacent pores and leads to the formation of pores wider (mesopores and macropores).

*Table 1:* Porous parameters of the activated carbons (impregnated carbons)

| | $S_{BET}$ ($m^2/g$) | $S_{mi}$ ($m^2/g$) | $S_e$ ($m^2/g$) | % Smi | Pores volumes($cm^3/g$) | | Pores sizes (nm) | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | $V_{micro}$ | $V_{cum}$ | BET $_{ads}$ | $BJH_{des}$ | $BJH_{ads}$ |
| OPSAC | 1442.49 | 1000.37 | 442.12 | 69.35 | 0.498 | 0.352 | 2.3967 | 4.7779 | 4.4546 |
| PETAC | 1063.46 | 753.88 | 309.60 | 70.89 | 0.335 | 0.5950 | 2.2380 | 4.1903 | 3.8736 |
| OPACI | 1221.7 | 312.6 | 909.1 | 25.59 | 0.1602 | 0.4909 | 2.2655 | 3.7693 | 3.7441 |
| PETACI | 1412.9 | 516.7 | 896.1 | 36,52 | 0.2634 | 0.4553 | 2.2085 | 3.7252 | 3.6253 |

*Table 2:* Porous parameters of the activated carbons (impregnated carbons)

| | $S_{BET}$ ($m^2/g$) | $S_L$ ($m^2/g$) | $S_{mi}$ ($m^2/g$) | $S_e$ ($m^2/g$) | Pores volumes($cm^3/g$) | | | Pores sizes (nm) | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | $V_{micro}$ | $V_{cum}$ | BET $_{ads}$ | $BJH_{des}$ | $BJH_{ads}$ |
| COM1 | 774.7 | | 632.21 | 168.58 | 81,61 | 0.251 | 0.3892 | 1.9838 | 3.0756 | 2.9440 |
| COM2 | 955.89 | | 714.41 | 241.47 | 74.74 | 0.315 | 0.4553 | 2.0551 | 3.8893 | 3.7054 |
| COM1I | 991.45 | 1227.8 | 829.93 | 161.52 | 83.71 | 0.3674 | 0.4574 | 1.8453 | 2.8920 | 2.8846 |
| COM2I | 1084.9 | 1274.5 | 570.26 | 514.7 | 52.56 | 0.2507 | 0.5439 | 2.0053 | 2.8875 | 2.9176 |

### b) *Adsorption in aqueous solution*

The methylene blue (MB) test was used to estimate the adsorption capacity of carbon to organic compounds from aqueous solution. The samples obtained showed good adsorption capacity for MB (Table 3). This indicated that all the samples were effective adsorbents and also accentuating the use of ACs in the elimination of large molecules in aqueous media particularly certain colorants [21]. For adsorption of $I_2$, important values were also obtained, which showed high micropores contained in the ACs samples (Table1). The adsorption in liquid phase shows consistency with the results found by the analysis bet. The adsorption values for $I_2$ for all the samples prepared by physical activation were between 930 and 1237 $mol.g^{-1}$ expressing the high adsorption capacities of the samples to smaller molecules indicating the presence of micropores.

However the adsorption of the BM is higher for the samples prepared by physicochemical activation than those obtained by physical activation, this shows the presence external pores (mesopores and macropores for instance).

The adsorption in liquid phase shows that physico-chemical activation produced a strongly developed internal surface, which is open at the surface by, mesopores and possibly macropores. This is important for the higher removal of both micropollutants and organic compounds due to the accessibility of internal pores sites [9, 22].

*Table 3:* Value of quantities of methylene blue and iodine adsorbed

| Activated carbon | OPSAC | OPSACI | PETAC | OPSACI | COM1 | COM1I | COM2 | COM2I |
|---|---|---|---|---|---|---|---|---|
| IBM (mg/g) | 317,26 | 472,42 | 288,37 | 460,03 | 455,58 | 493,02 | 215,92 | 334,88 |
| I$I_2$(mg/g) | 930,23 | 827,91 | 1110,41 | 567,44 | 1237,31 | 348,99 | 1221,45 | 380,26 |

### c) *Analysis of Fourier Transform Infrared Spectroscopy (FTIR)*

To see the effects of KOH on the impregnation of carbonized material, we need to compare the spectrum of a sample obtained by physical activation in Figure 4 to that obtained by physicochemical activation in Figure 5. The frequency range studied is between 400 and 4000 cm$^{-1}$, it can provide valuable information about the functional groups on the surface of the materials. These spectra show three main absorption peaks at about 1097 cm$^{-1}$, 1660 cm$^{-1}$, 3391 cm$^{-1}$. From this comparison, it is clear that the bands of samples obtained by physicochemical activation are more intense than those prepared by physical activation, this simply demonstrates that the impregnation of the samples with KOH during physicochemical enhance the surface functions. The intense vibration found at 3391 is due to the existence of hydroxyl groups on the surface, and of the adsorbed water, the absorption peak around 1120 cm$^{-1}$ indicated the existence of C=O bond stretch. The absorption peak around 1600 cm$^{-1}$ can be attributed to both C=C aromatics cycles and carbonyl C=O stretching vibration; this zone justifies the fact that there is not graphitization during the carbonization process of all samples which is favorable for the formation of porosity into the structure of the carbon structure [20].

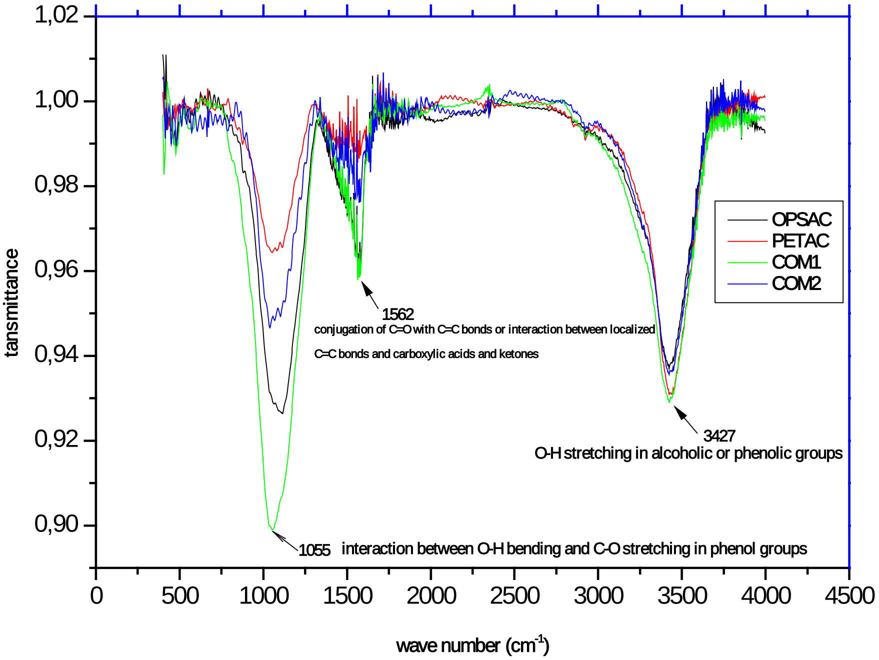


*Figure 3:* FTIR spectra of four activated Carbons obtained from both oil palm shell and post-consumer plastics using physical activation method with steam

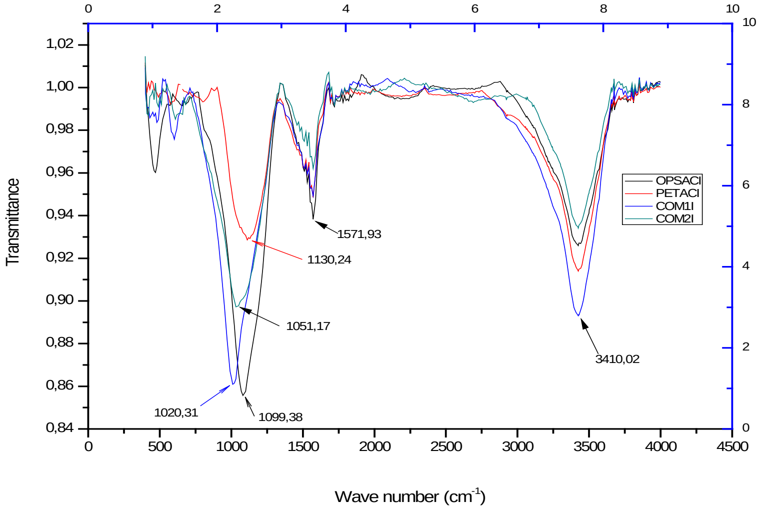


*Figure 4:* FTIR spectra of four activated Carbons obtained from both oil palm shell and post-consumer plastics using physicochemical activation method with steam and KOH

*d) Raman spectroscopy*

Raman spectroscopy has been an important tool in such investigations because it allows to a better understanding of the microstructure of the carbon (Yan Wang). The Raman spectrum reveals disordered graphitic materials suggested by the two Raman modes the peaks at 1590 cm$^{-1}$ (G-band) corresponds to and $E_{2g}$ mode of hexagonal graphite and is related to the vibration of sp$^2$ hybridized carbon atoms in a graphite layer which means that materials are composed of graphitic carbon.

The D band at about 1360 cm$^{-1}$result from large defects within a carbon network, it is the vibration of carbon atoms with dangling bonds in the plane termination of disordered graphite. The relative Raman intensity of the D and G bands is, therefore, a measure of the degree of order in amorphous carbon samples (Yan Wang). We observed that the intensity of the G and D bands enhance when the carbonaceous material is impregnated with KOH. This confirms that impregnation of carbon increase lattices into carbon material and then developed the internal network of AC obtained [22,23].

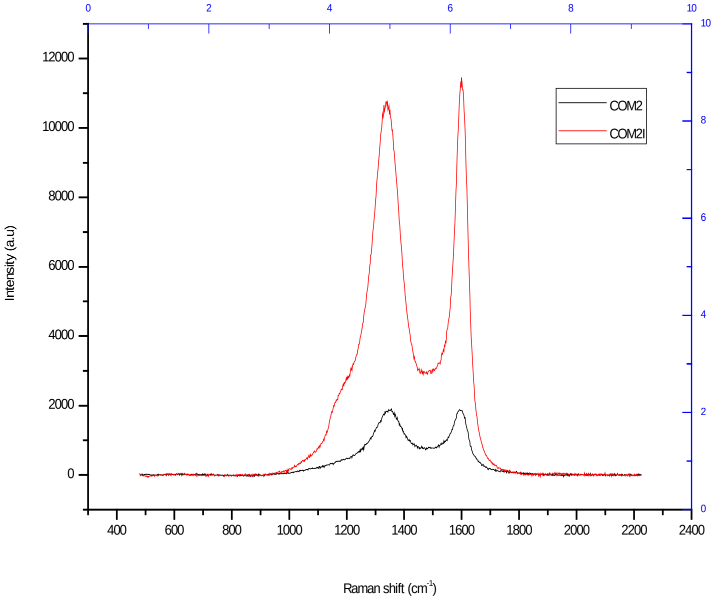


*Figure 5:* The Raman spectra of ACs obtained by mixing lignocellulosic material and post-consumer plasticsin the ratio 1:2, COM2 is prepared by physical activation with steam, COM2I is prepared by physicochemical activation, char is impregnated by KOH before activation under steam at 800°C

*e) X-ray diffraction*

The X-ray diffraction (XRD) patterns of ACs obtained from the same precursor by physical activation and physico-chemical activation COM2, and COM2I are shown in figure X. The sharp diffraction peak in the COM2I sample appearing at 2θ = 20, 98°; 2θ = 50,44°; 2θ = 67,5°, 2θ = 73,9° may be attributed to the presence of ash due to the impregnation of char by KOH before activation process with steam. However, these peaks disappeared from the sample prepared by physical activation COM2. The XRD patterns of COM2 and COM2I exhibit in Figure X respectively peaks at 2θ=36,06°, 2θ=41,3° attributed to the turbostratic structure of carbon materials, the peak intensity increased significantly for the sample obtained by physical activation, which indicated an increase graphitization in the structure of carbon material [11,22].

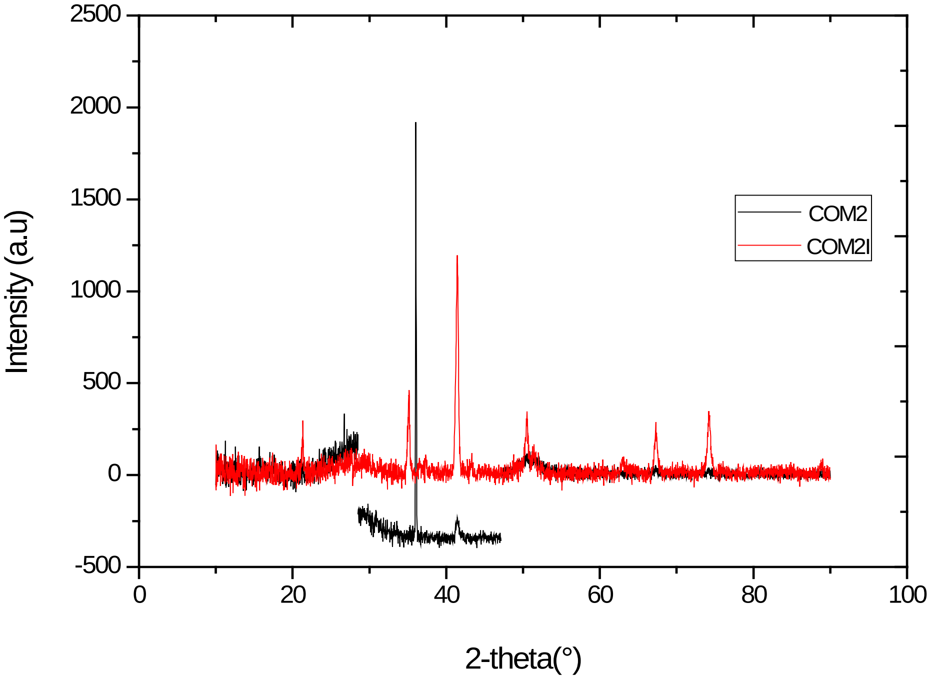
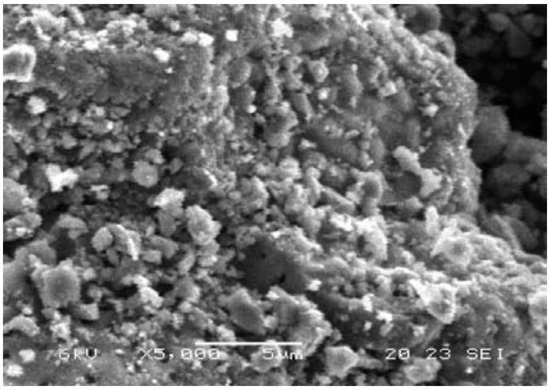


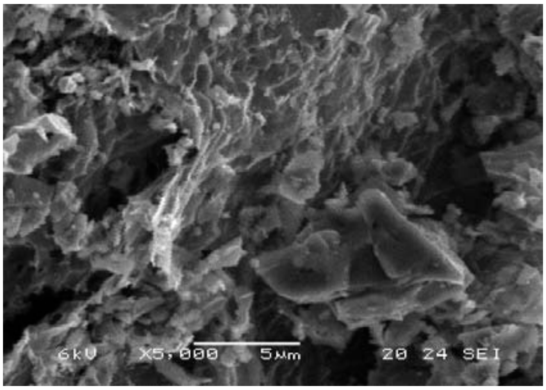
*Figure 6:* XRD patterns of ACs obtained by mixing lignocellulosic material and post-consumer plastics, COM2 is prepared by physical activation with steam, COM2I is prepared by physicochemical activation char is impregnated by KOH before activation under steam

*f) Scanning electron microscopy of activated carbon*

Scanning electron microscopy (SEM) is used to observe better the microstructure of the activated carbons prepared. The micrographs obtained are shown in FIGS.



(a)



(b)

*Figure 7:* SEM images of a) COM2 is prepared by physical activation with steam, b) COM2I is prepared by physic-chemical activation, and char is impregnated by KOH before activation under steam at 800°C

Examination of these images reveals that independently of the composition of raw material the entire sample studied are characterized by aggregates of different sizes and irregular shapes. For sample prepared by physical activation, small pores are observed over the entire surface of the samples, the for samples prepared by physicochemical activation we can observed pores diameter ranging from 0 to 5 µm which give to a higher developed network porosity inside the carbon samples [11, 22].

## IV. CONCLUSION

Activated carbons were prepared from lignocellulosic material and post-consumer plastic by combining physical and chemical activation named physicochemical activation, the same precursor was subjected to physical activation for comparison. The results showed that samples obtained by physical activation are essentially microporous with a surface area between 1000 and 1500 $m^2/g$. Physicochemical activation leads to the higher development of porosity of ACs by transforming micropores at the surface to mesopores. The adsorption test in aqueous solution shows an affinity of four samples prepared by physicochemical activation to methylene blue, which show external mesopores which connected external surface to the internal pores. FTIR spectrum of ACs prepared shows many peaks belonging different functional groups, samples obtained by physico-chemical activation give peaks more intense than those obtained by physical activation showing that they are more functional group at the surface of ACs obtained by physicochemical activation. Raman and DRX spectrum show that impregnation of char increase lattice into carbon material and then developed the internal network of AC obtained, It was found in this study that physicochemical activation allows obtaining ACs with higher developed porosity constituted of micropores, mesopores, and likely macropores.

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