Nanostructured Carbonaceous Porous Material Synthesis

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August 7, 2021

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***Abstract*—Nanostructured carbonaceous porous materials based biomasses were prepared through thermal and/or KOH chemical activation using potato peels. It was then characterized by XRD, Raman, FESEM FTIR and BET surface analysis. The semi-amorphous character of the produced carbon material was confirmed by XRD patterns and Raman spectra in comparison to the raw material structure. The FESEM images and EDX analysis revealed the formation of heterogeneous mesopores with some inorganic traces on the surface of the nanostructured materials with BET specific surface area around 2030 m^2/g, 1.30 m^2/g for PP1/3 and PP1/0, respectively. Furthermore, we have demonstrated that synthesis conditions and spatially activation process have significant impact on the textural properties and O-based functional groups.**

Keywords—Nano-structure material, Activated Biochar, potatoes peels, Biomass, Pyrolysis.

I. INTRODUCTION

Nowadays, the primary goal of all research is to generate new technologies that will improve the quality of life for the general public. Nano-materials have recently become the most appealing smart materials used in current technology, yet they are still regarded costly. Thus, various studies have been conducted with the goal of converting Biomass into nanostructured material, where this technology not only solves the problem of waste disposal, but also transforms potentially waste product into a valuable product that can be used in a variety of applications [1]. Among them, the nanostructure porous carbonaceous material as know activated biochar is characterized by highly developed pore texture, specific area and surface functionalities [2]. These entire features can be enhanced by synthesis parameters.

The main objective of the present study is to produce porous carbonaceous material from potatoes peels waste biomass with very important properties.

II. MATERIEL AND METHODS

A. Preparation of activated Biochar

Activated biochar from potato peels were carried out by tow kind of synthesis process, the dried biomass was ground by a coffee grinder; the latter was denoted as PP and has undergone a thermal treatment into a horizontal quartz reactor of furnace CARBOLITE under nitrogen inert atmosphere. The carbonized product referred to PP0. In order to show the difference between thermal and thermo-chemical activation process, carbonized crudee potato peels was activated using potassium hydroxide at 600°C. After that, the obtained sample was immersed in 2M HCl to remove any inorganic salts, and then washed several time with distilled water until a neutral pH (6-7). Finally, the final product was dried in an oven overnight at 80°C, samples were labeled as **PP0, PP3.**

B. Activated biochar characterization

The structural, morphology, textural properties of samples were analyzed using X-ray diffraction (XRD), Raman analysis, Field Emission Scanning Electron Microscopy (FESEM) and EDX, elemental analysis CHNS-O and N_2 physical adsorption/desorption BET.

III. RESULTS AND DISCUSSION

High resolution scanning electron microscopy (FESEM) is frequently used to analyze surface morphology, including pores structure and distribution. Fig. 1, shows surface morphology of native potato peels. The surface of the potato peel comprises irregular starch particles with an elliptic or spherical shape [3]. On the other hand, the FESEM micrographs of PP0 and PP3 showed a significant modification in comparison with starting material PP. They showed mesoporous cavities on the surface of treated samples. It is clearly shown that the carbon surfaces were highly porous nature in PP, which confirms the effectiveness of thermo chemical process in producing nanostructured carbon materials.

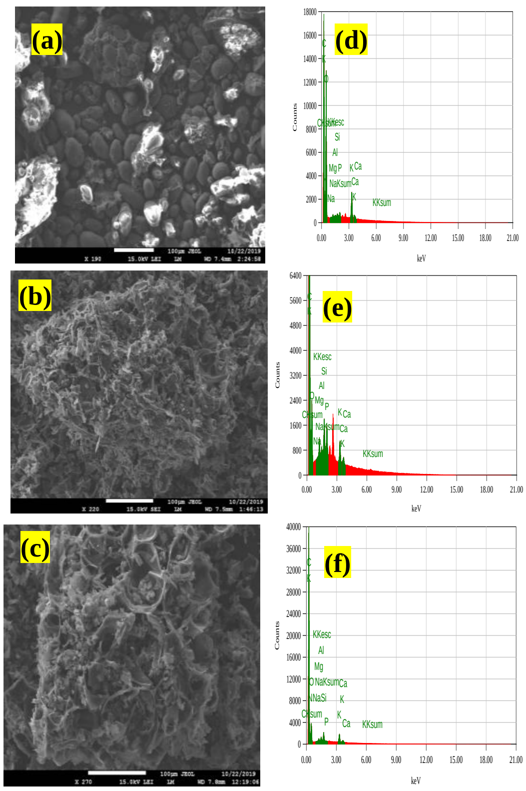

Fig. 1. FESEM images (left) of (a) Native potato peels (PP), (b) carbonized Native potato peels (PP0), (c) Activated carbon (PP3). Their EDX (right) (d), (e), and (f).

The EDX analysis as reported in Fig. 1 of these samples indicates that all the products contained several elements such as: carbon, oxygen, nitrogen and other inorganic elements such as : **Mg, P, Si, Al, Ca, Cl and K** [4].

Tab. 1, represent the results of elemental analysis which indicated the percentage of carbon, hydrogen, nitrogen and sulphur present in the sample. The study suggested that the principal components inside activated carbon are oxygen and carbon. As a result, our biomass is particularly suited as a raw material for producing activated Biochar [5]. The carbon content of the product PP0 after thermo or thermochemical activation increased and became higher, while H, O, N and S gradually reduced in comparison to the biomass. The decrease in H, O element content may be attributed to the dehydration and decarboxylation reactions during the decomposition of biomass, evolving out H_2, CH_4, CO, CO_2 as gas products. [6]. Moreover, the carbon and oxygen content were reduced after thermo-chemical activation due to the chemical reaction between carbon and KOH activating agent, which leading to the oxidation of carbon generating more CO and/or CO_2 [7].

TABLE 1. CHNS-O elemental analysis of samples.

Samples	**Elemental composition %**				
	C	**H**	**N**	**S**	**O**
PP	**38,16**	**5,55**	**1,16**	**0,03**	**42,94**
PP0	**59,53**	**0,89**	**2,03**	**0,2**	**15,27**
PP3	**57,04**	**1,17**	**0,49**	**-**	**12,44**

Using HighScore Plus 3.0d (3.0.4), it was possible to identify amorphous and crystalline structure of all samples. X-ray diffraction spectra of samples were shown in Fig. 2.

The XRD pattern of untreated potato peels showed many diffraction peaks at 17.02°, 22.11°, 19.5°, 24.11° and 26.56° , which are attributed to the essential compound in potato peels such as starch and cellulose compounds [8].

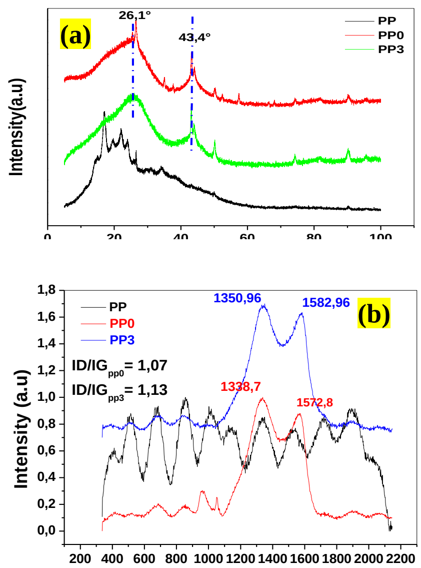

Fig. 2. XRD patterns (a) and Raman analysis spectra (b) of Native potato peels (PP), carbonized Native potato peels (PP0) and thermo-chemical activated crudee potato peels (PP3).

All these characteristic peaks were disappeared after thermal and / or chemical treatment due to the breakdown of the starch chain or cellulose structure with the variation in chemical composition under such conditions [9]. The structure of activated biochar became more and more amorphous under thermo-chemical activation process; which is the main favorable properties for promising activated biochar.

After such activation process, the XRD patterns of PP0 and PP3 show two broad peaks at 26.1° and 43.4°. They correspond the (002) and (100) graphite diffractions, indicating that both materials have aromatic layers arranged in a turbostratic structure [10].
Moreover, the presence of sharp and strong peaks in all samples was correlated to inorganic complex such as silicon which is originated elements of started material [11]. The intensity of these sharp peaks for PP3 is lower than PP0, which illustrates the positive effect of washing steps to remove such inorganic elements and improve therefore the structure quality.

To confirm the structural characteristics of these products, the Raman spectra were obtained and their spectra are shown two dominant bands at 1350 cm^{-1}, 1338 cm^{-1} (D-band) and at 1582 cm^{-1}, 1572 cm^{-1} (G-band) for PP0 and PP3, respectively. Moreover, the intensity ratio of D and G bands intensity (ID/IG) of PP0 and PP3 is 1.04, and 1.13, respectively; indicating that PP3 was more amorphous structure which prove x-ray diffraction results [12].

The functional groups on the surface of products were determined by FTIR spectroscopy. The FTIR spectrum is shown in Fig. 3, for PP the band centered at 3400 cm^{-1} which can be ascribed to O-H stretching vibrations in alcohol, phenol, and carboxylic groups from the phenol groups of hemi-cellulose, cellulose and lignin or/and N-H stretching vibration [13].

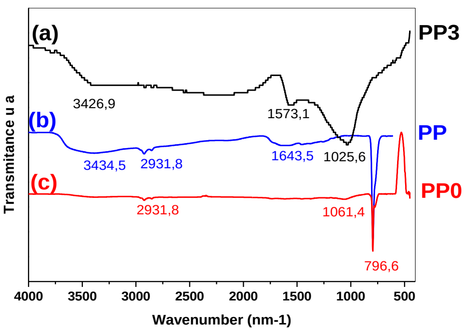

Fig. 3. FTIR of Native potato peels (PP), carbonized Native potato peels (PP0), thermochemical crudee potato peels (PP3).

After thermal and /or chemical activation, the spectrum shows one other peak located at around 1650 cm^{-1}, ascribing to the presence of C=C stretch from carbohydrates, carboxylic groups –COOH[14]. The sharp peaks appearing after thermal activation process over 1020-1060 cm^{-1} and 797.6 cm^{-1} are assigned to Si-C stretching vibration or heteroatomic element such as Si [15] and plane bending of aromatic compounds ring C–H bonds [16], [17].

In order to estimate the type of pores present in our products, we have carried out their isotherm curves of native potato peels, and crude potato peels thermal activation PP0 compared to thermo-chemical activated crude potato peels PP3 Fig. 4 The adsorption-desorption isotherms for crude potatoes peels does not according to any type of model existed in literature, which confirm the non porous nature of crude potato peels biomass with very low specific surface area. After thermal activation of crude potatoes peels, the textural property of starting samples was enhanced, as shown by the adsorption-desorption type IV. Therefore, the N_2 adsorption was carried out at very low relative pressure *P/Po* which is attributed to its microporosity; similar result was obtained by Thangavelu Krithiga and co-workers [18].

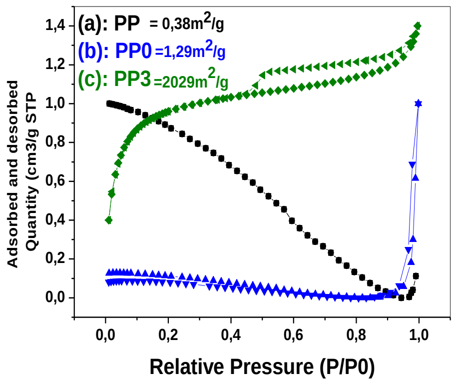

Fig. 4 . N_2 (77 °K) adsorption-desorption isotherms of (a) Native potato peels (PP), (b) carbonized Native potato peels (PP0) and (c) thermo-chemical activated crude potato peels (PP3).

According to literature, Isotherm type II profile of PP3 presents a hysteresis loop of type H4 at P/P0 of 0.4 associated to narrow slit-like shape.
In comparison to the crude potatoes peels thermal activation the hysteresis loop became wider, indicating the presence of more mesoporous proportion [19]. The obtained result was confirmed by their above obtained morphology.

IV CONCLUSION

In conclusion, we have investigated the effect of synthesis strategy in developing nanostructured carbonaceous materials with interesting properties. The overall nanostructured material derived waste biomass produced through KOH chemical activation possessed the most promising properies (morphology, textural and functional groups) suitble for several applications such as energy storage, wastewater treatment, sensor, catalysis, ...etc.

Therefore, the nanostructured material prepared actually via new KOH chemical activation exhibit higher specific surface area compared with the such prepared via thermal process. Morphology analysis associated by EDX analysis confirmed the porous nature of our carbonaceous materials, with some trace of elements originated of native biomass. XRD and Raman studies suggested the semi-amourphous nature of these nanostructured materials.

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