Physico-Chemical Properties of Three Synthesized Carbonaceous Nanomaterials (CNTs, GO, Biochar) for Perspective Application: Water / Soil Treatment and Energy Storage

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Abstract− Nanotechnology has a more than important role in deducing the materials' structure, especially carbon-based nanomaterials, including determining their properties and consequently their application field, such as: energy storage, environmental protection, biosensing and soil treatment. In this investigation, we report a comparison of three kinds of nanostructured carbon based smart nanomaterials synthesized at different dimension (1D, 2D and 3D) using physico-chemical growth processes. These carbonaceous products have been characterized in order to identify their attractive properties using different techniques, such as XRD, FT-IR, TGA/DTA, FESEM microscopy, Raman and XSP spectroscopy. These obtained nanostructured carbon have shown structural forms in the case of MWNTs and graphene type having 1D and 2D configuration, respectively, as well as an amorphous form in the case of biochar having 3D porous configuration which contains less cohesive bonds than graphene and MWNTs. These two structured ones have a much more solid and cohesive structure thanks to the strength of their carbon bonds and their graphitization rate is proved from their Raman and XPS-C1s analysis spectra. Moreover, they have shown very interesting characteristics especially their specific surface area in the range 150-2400 m^2/g and functional groups; which open up a wide field of application especially environmental protection and biosensing.

Keywords− Carbonaceous nanomaterials, Carbon nanotube (CNTs), graphene**,** biochar**, Environmental protection Soil treatment.**

I. Introduction

Humanity lives today under serious threats which negatively affect its various activities. Pollution can be considered as the first concern and the main cause of problems related to most areas including health and economics. So, it is necessary to rely on natural, renewable and environmental friendly sources as the main solution to these issues. This is in parallel with the work on the technological development of energy storage, depollution, sensing, which prompted the world to fight against these anomalies thanks to the emergence of Nanosciences and Nanotechnologies [1-5].

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Nanotechnology is most important at the forefront of today and exciting fields, such as materials science, engineering, biotechnology, agriculture, etc. It has given great hope for scientific revolutions in the near future that have change the direction of technology in many application fields. The concept of nanotechnology is based on the assumption that smaller particles than one hundred nanometers give the material new attractive properties and new behaviors which express new physical and chemical concepts [1, 6, 7].

Nanotechnology has opened the way to study the matter shapes and the components at the nano level (atomic scale). Moreover, nanomaterials are one of the main nanotechnology products that can be categorized into three types: one-dimensional (1D), two dimensional (2D) and three dimensional (3D) where each of these materials exhibits unique properties based on its particular characteristics [8, 9]. Among the most important of them, we mentioned the carbon nanotubes (CNTs) which are composed either of a single sheet of graphene (SWNTs) rolled up on itself in such a way to form a cylinder (1D) with a diameter between 0.4 and 3 nm; or multi walled nanotubes (MWNTs) with inter-tubes distance 0.34 nm [10-14]. Then, we find graphene which is the thinnest and strongest material known to date. It is a 2D crystalline sheet, as the first studied crystal of allotropy, based on hexagons of carbon one atom thick in sp2 hybridation in a honeycomb structure, at the origin of Graphite. More precisely, FLG (Few Layer Graphene) have emerged as potential platforms for exploring novel and unique properties no found in other materials due to their planar geometry and their related in-plane propagation properties [15]. Finally, biochar is the solid part obtained through pyrolysis of the organic matter biomass in the absence of oxygen [16, 17]. It possesses amorphous structure in 3D configuration with interesting contact specific surface [16, 18].

In this paper, we present the successful synthesized products based on three kinds of carbon-based nanomaterials (biochar, MWNTs and graphene) and the comparison between them using very specific characterization techniques to deduce their attractive physico-chemical properties. They have shown very interesting characteristics basing on specific surface area and functional groups which open up to a wide field of their application as smart nanomaterial.

II. Experimental Procedure

A. Carbonaceous nanomaterials synthesis

Biochar production: Activated biochar from potato peels biomass was prepared through double pyrolysis process basing on chemical and physical aspect. At first, the potato peels, with a thickness not more than 2 mm, were cleaned and natural dried for 48 hours. After that, the dried biomass was ground using Coffee grinder to obtain very fine particles. Then, 20 g of this dried matter with and without oxidant agent was placed in a ceramic boat and transferred onto horizontal quartz reactor inside the cylinder furnace under an inert N_2 atmosphere (200 ml/min). Therefore, the pyrolysis is carried out via a simple thermal cycle with heating speed 5°C.min^{-1} and carbonization temperature of 600 °C during 2 hours. Consequently, the produced activated biochar was immersed in 2 M HCl for 24 h to remove any inorganic salts and then washed several times with distilled water until a neutral pH achievement [16].

MWNTs par CCVD: The MWNTs studied in this investigation were synthesized using catalytic CVD technique over using Fe/Al_2O_3 catalyst support at 750 °C growth temperature during 1 hour with acetylene and nitrogen as carbon source and carrier gas, respectively. After that, they were purified and functionalized through heated acid treatment (HCl : HNO_3 = 1:3) at 80 °C for 2 hours, then washed and filtered several times with deionized water until no detected residual acid. Finally, they were dried at 80 °C for 20 hours [10].

Graphene exfoliation: Direct liquid-phase exfoliation of layered materials by means of ultrasound was used to synthesis few layered graphene (FLG) using Janowska team process [19].

B. Characterization techniques

For structural identification, these carbonaceous materials were characterized with powder X-ray diffraction (PXRD) (PAN analytical XPert PRO Bragg–Brentano diffractometer) equipped with CuKα (λ = 1.540598 Å) where the analysis was performed with a step size of 0.017°.min^{-1} at room temperature in the 2θ range [5 - 90°]. The relative crystallinity degree (CD) was estimated using following equation [16], where Ac and Aa are the area of the crystalline and amorphous peaks, respectively, in the XRD diffractogram. $CD\ (\%) = \frac{Ac}{(Ac + Aa)}$.

Moreover, the FT-IR technique is an important tool also to identify the chemical binding of formed functional groups. We have used an analyzed by a Bruker Vertex 77 v spectrometer with a 4 cm^{-1} resolution controlled with an Opus software in the wave number range of 4000 − 400 cm^{-1}. In addition, Raman analysis was used to identify quantitatively and qualitatively the carbon-based nanomaterials in order to understand their crystalline/defect nature. So, we have carried out this analysis on a Horiba Jobin Yvon Lab – Ram Aramis confocal Raman Spectrometer recorded using 532 nm laser excitation with 0.33 mW power. Thermal analysis measurements have been also carried out using the TGA/DTA technique which gives qualitative information on the products composition as well as their percentage as quantitative estimation. Therefore, we have used the TGA 550-TA instrument with a heating speed of 10 °C.min^{-1} from room temperature to 1000 °C under an air atmosphere. Besides, the surface morphology of these products was analyzed using a Field Emission Scanning Electron Microscopy "FESEM microscopy" (JEOL JSM-7500 F Model) equipped with a secondary electrons detector with 15 kV accelerating voltage showing the textural morphology. The specific surface area (SSA), total pore volume and pore size distribution as important textural characteristic for ulterior use of produced carbonaceous nanomaterials were carried out via N_2 adsorption–desorption isotherm (at 77 °K liq-N_2) using ASap2020 Micrometrics. Also, the Barrett–Joyner–Halenda (BJH) and Density Functional Theory (DFT) methods were used automatically to provide a micro- and meso-scopic pores distribution, respectively. At last, X-ray induced photoelectron spectroscopy (XPS) is a method of surface analysis which allows to thoroughly determine the chemical composition and binding of the studied products. In this investigation, we focalize on the functional groups information and the bonds nature to follow the chemical surface modifications. So, we have used Scientific K-Alpha Spectrophotometer by monochromatic Al Kα radiation (1486.6 eV). First, a wide sweep scan was carried out from 0 to 1400 eV as survey scan to define the main element present in the product. After that, the main peak precise analysis to obtain base-level spectra has been calibrated with respect to graphite carbon where C1s was leveled at 284.6 eV. Composition products and their main peaks deconvolution for all reached XPS spectra were performed using Avantage softwar where the curves fitting has been done basing on mixed Gaussian/Lorentzian peak shape (by 0.3).

III. Results and Discussion

Structural Characteristics

Figure 1 illustrates the typical XRD patterns of synthesized biochar and MWNTs at optimized conditions. This structural identification of these products through their characteristic peaks using JCPDS reference cards allows differentiating them from a structural aspect point of view.

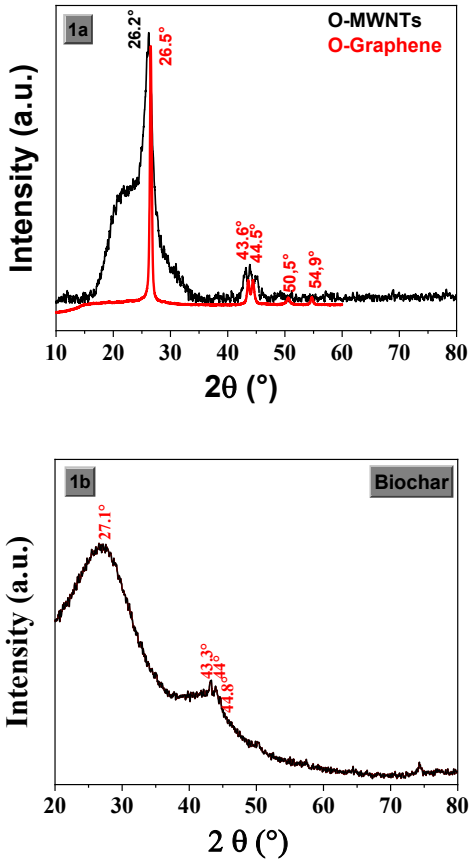

Fig. 1: XRD diffractograms of two different carbonaceous nanomaterial: MWNTs and biochar.

Therefore, the XRD spectrum (Fig. 1a) confirms the formation of carbon nanotubes "CNTs" with an hexagonal structure according to the JCPDS reference card N° [00-041-1487] through narrow shape peaks located at 26.3°, 43.2°, 43.9° and 45° corresponding respectively the following crystalline plans (002), (100), (101) and (101) [10]. Also, the more intense diffraction peak was observed at around 26.3 (MWNTs) - 26,5° (graphene) corresponding to (002) plan with an interlayer spacing of about 0.34 nm [20].

However, the spectrum in figure 1b is characterized by almost the same 2θ positions with some shifts confirming the carbon composition but with broader shape aspect than those of CNTs indicating the product semi-crystalline state [16].

So, this structural identification confirms clearly the amorphous aspect of produced biochar in comparison to structural one (Fig. 1a) that was proved from their estimated crystallinity degree (CD). We found MWNTs with more crystallinity (42 %) than biochar (5.6 %) justifying consequently more the below results.

To identify more exactly the main existing chemical bands inside these products, figure 2 represents their obtained FT-IR spectra to reveal their functional groups in the range [4000 – 400 cm^{-1}]. Generally, the types of chemical binding can be classified according to their wave number values. In these products focalizing on the strong broad peaks, we can observe that in the case of MWNTs, the main binding bands are assigned to : 1553 cm^{-1} (C=O / C-O), 1648-1694 cm^{-1} (C=C), 3668-3855 cm^{-1} (O-H) and 2850- 2926 cm^{-1} (CH_3/CH_2) [20, 21]. Whereas, in the case of biochar, the essential bands are found at 3163 cm^{-1} (COOH and –COH), 1586 cm^{-1} (C=O), 1442-1350 cm^{-1} (carbonate ions) and 2143-2035 cm^{-1} (O-H).

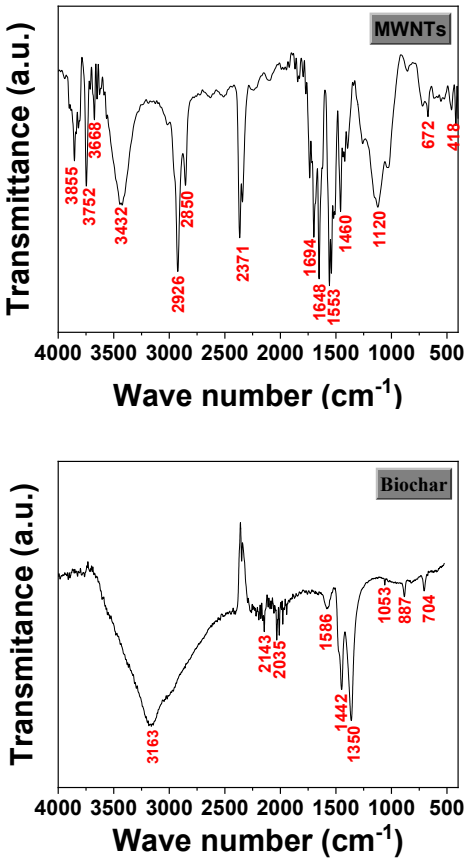

Fig. 2: FT-IR spectra of two different carbonaceous nanomaterial: MWNTs and biochar.

Thereby, according to these differences in bonds and consequently the nature of the atoms binding, we can easily deduce that MWNTs are more structured, rigid and coherent by comparing them with biochar product, which is much more based on the carbon-oxygen binding bands.

From their Raman analysis as shown in figure 3, we clearly observe the unique and essential two peaks "D" and "G" bands characterizing the carbonaceous materials [10, 22]. Usually, the G band peak gives the information on the graphitization degree through its position and intensity.

Its position is on a high Raman shift value in the case of crystallized nanomaterials with more graphitization which is found more with MWNTs (at 1596 cm^{-1}) and graphene (at 1580 cm^{-1}); while it is on its lower value (at 1566 cm^{-1}) in the case of amorphous carbon ‘’biochar’’.

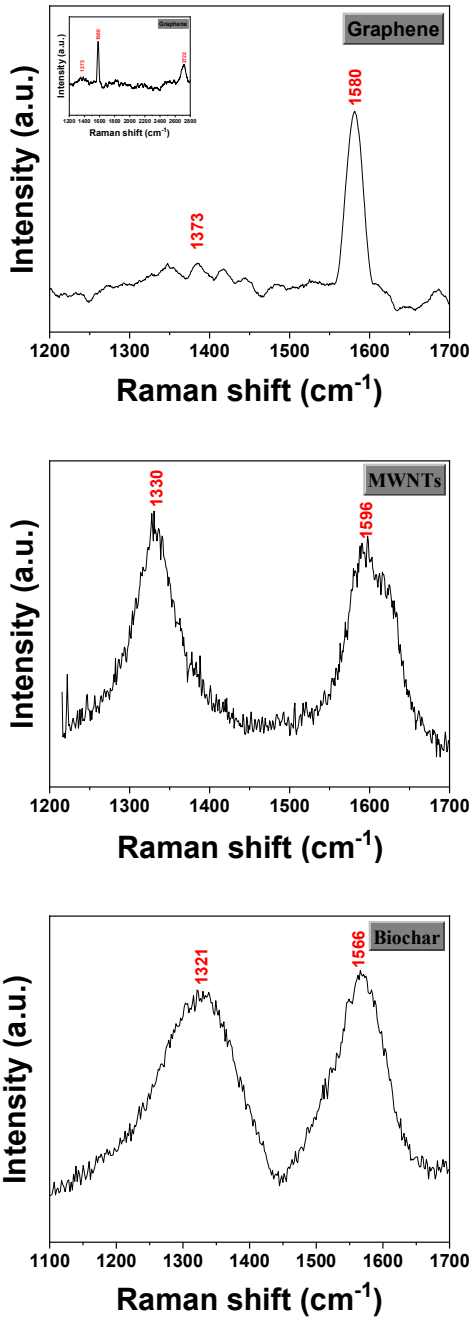

Fig. 3: Raman spectra of three kind of carbonaceous nanomaterials: graphene, MWNTs and biochar.

However, the D band peak shows the existence of defects in the carbon-based lattice of these products. Their values are found around 1321 cm^{-1} for the amorphous biochar and 1330 or 1384 cm^{-1} for the structured carbon like MWNTs and graphene, respectively [22]. According to these Raman spectra for three

studied carbon based products (MWNTs, graphene and biochar), we regroup in the following table I the deduced characteristics which confirm the structural/graphitization and defect difference.

Consequently, we clearly confirm that the much more graphitized product was the graphene and the MWNTs with estimated graphitization ratio I_G/I_D around 3.6 and 1.11, respectively. While, the disordered product was found with biochar proving by their estimated defect/graphitization I_D/I_G

Table. I
THE CHARACTERISTICS OF THE RAMAN PEAKS FOUND WITH THESE THREE KIND OF STUDIED CARBON-BASED NANOMATERIALS.

Samples	Position D (cm^{-1})	Position G (cm^{-1})	I_D/I_G	I_G/I_D
biochar	**1321**	**1566**	**0.93**	**1.08**
MWNTs	**1330**	**1597**	**0.90**	**1.11**
graphene	**1384**	**1580**	**0.28**	**3.60**

intensities ratio around 0.93. Moreover, this means that the graphene structure contains low impurities and defects compared to other studied carbonaceous nanomaterials (MWNTs and biochar) in this investigation [21, 23].

Therefore, from these intensities ratio, we recorded the graphitization following order graphene < MWNTs < biochar.

In addition, to estimate the graphene layers number we focalize on the D and 2D peaks as reported by V. Kumar work [15]. We found intensity ratio $I_D/I_G = 0{,}28$ and $I_{2D}/I_G = 0{,}54$ with 2D peak position at 2720 cm^{-1}; which confirm the obtaining of 7 to 10 graphene layers proving hence Few Layer Graphene (FLG) based product.

Thermal Characteristics

In order to confirm our previous results, we have also used a thermal analysis (TA) technique which allows obtaining the thermal properties of these studied products. This technique gives qualitative/quantitative information on nature and composition of these products. Figure 4 illustrates two spectra of this carbonaceous nanomaterials thermal analysis. In the case of biochar, we observe that there are mainly four steps on the mass loss curve (TGA) and its equivalent in four oxidation peaks obtained on their derivative curves (DTA). These mass losses are characterized by their oxidation/combustion temperature found at: 30 °C, 156°C, 411°C and 616°C, respectively. The first 12 wt.% loss was attributed to moisture loss; while the second loss was found with 8 wt.% which is due to the presence of adsorbed water. However, the third loss which is dominant with a percentage 65 wt.% presents the transformation and the combustion of their carbon-based skeleton into CO_2. Finally, a last slight loss of 5 wt.% can be attributed to the presence of a structured carbon network. Therefore, the overall mass loss for this biochar as amorphous carbon based product is 90 wt.% with a residue of 10 wt.% due to a some residual traces of metals from the used biomass [16].

In the same way for the case of MWNTs as nanostructured carbon product, we observe that there are mainly two oxidation peaks found at 238°C and 615°C, corresponding to two losses of 5 wt.% and 90 wt.%, respectively. Consequently, the MWNTs possess a high purity and selectivity of 95% with residues of 5 wt.% that represent the used catalyst during their CCVD growth [10]. Finally, this thermal analysis allows to distinguish that MWNTs are more thermally resistant (with T_{oxy} = 615 °C) compared to biochar with a slightly lower oxidation temperature (T_{oxy} = 412 °C) proving the difference of their carbon network and the defect existence inside this network.

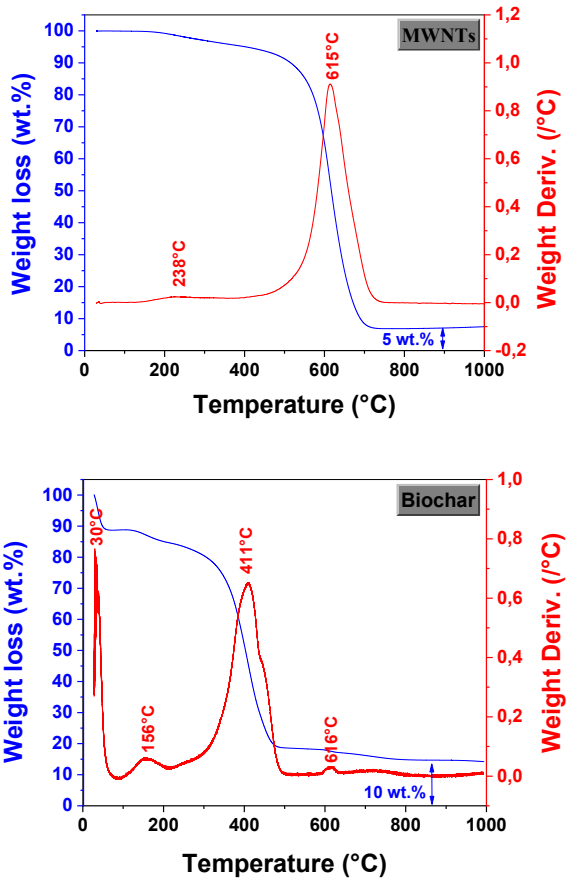

Fig. 4: Thermal analysis (TGA/DTA) of studied amorphous biochar and nanostructured MWNTs.

Morphological properties

FESEM microscopy is widely used to determine the surface morphology including the textural aspect of these carbonaceous products as illustrate in figure 5. Figure 5a shows the acquisition of one-dimensional (1D) tubular shapes with more or less homogeneous diameters between 20 and 50 nm. Their high resolution TEM (Fig. 5b) confirms that these nanotubes clearly are composed of multi walls cylinders which prove the Multi-Walled Nanotubes (MWNTs) formation. These MWNTs have average diameter around 20 nm with canal diameter around 10 nm and 14 walls possessing 0.36 nm calculated graphitic inter-crystallographic plan distance as shown inset figure 5b [10].

This kind of carbon nanotube is more useful for many application fields due to their contact surface which is around 150 m^2/g that can be provided via the channels and voids between plans as well as their functional groups. However, figure 5c illustrates very thin 2D shape with a nanometric thickness. It confirms that the resulting graphene has a relatively smooth planar structure which tends to overlap. These graphene sheets have few layers as confirmed through the Raman results from 2D peak form.

In contrast to these structural carbons based products, biochar as represented in figure 5d, shows an amorphous spongy textural morphology of porous structure with relatively irregular surfaces containing many cracks (pores) [24]. This morphology gives it an interesting compositional textural aspect with a very interesting contact surface which found approximately around 2400 m^2/g [16].

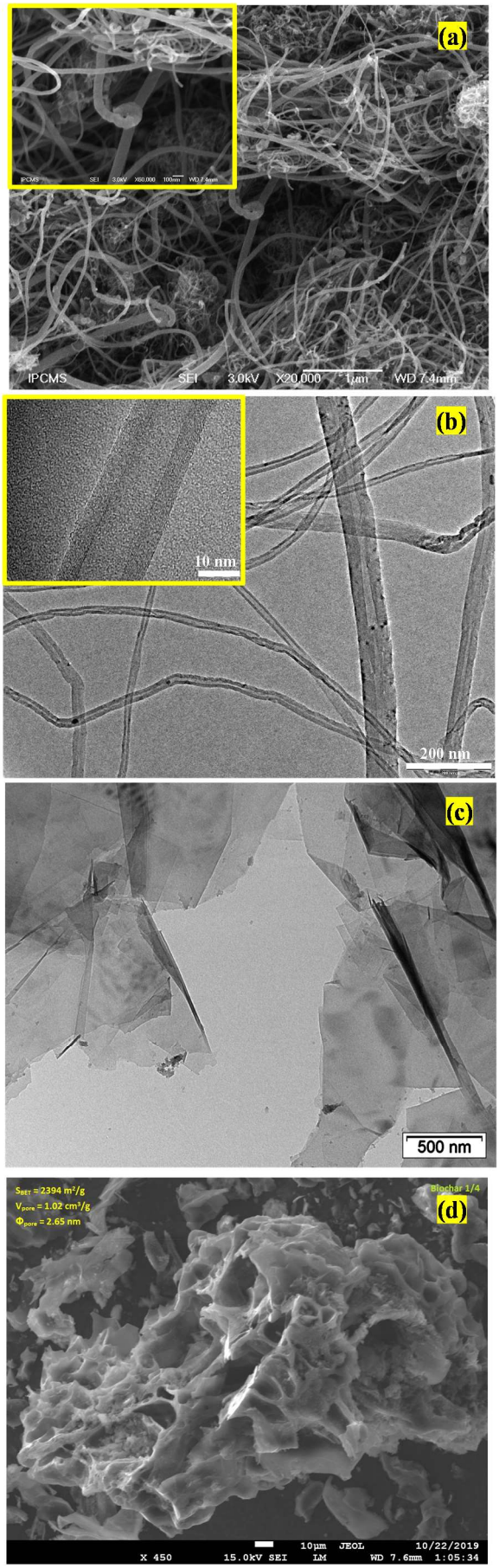

Fig. 5: High resolution FESEM and TEM micrographs for three kind of carbonaceous nanomaterial.

The above estimated specific surface area (contact surface) values of synthesized MWNTs and biochar have been performed through BET textural analysis as reported in figure 6. The N_2 adsorption–desorption isotherms (Fig. 6 left) can be described as a combination of type I and type IV isotherms. Typically, these isotherms show an apparent hysteresis loop indicating the presence of mesopores accompanied with micropores which are confirmed by the pore size distribution inside products [16]. Moreover, as can be seen MWNTs are only based of mesoporous structure (pore size [2 – 50 nm]) while biochar have dominant micropores (< 2nm) with some mesopores around 4 nm, as shown in pore size BJH distribution curves (Fig. 6 right).

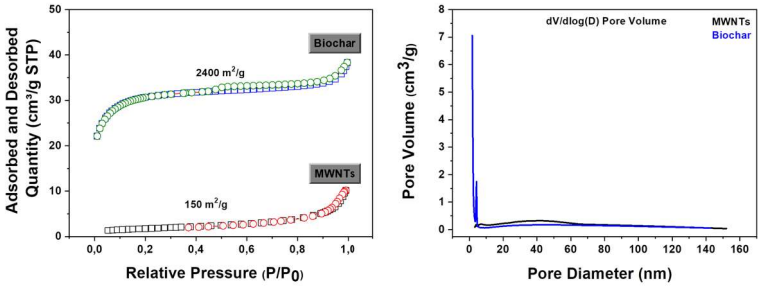

Fig. 6: N_2 adsorption–desorption isotherms (left) and their corresponding pore size distributions (right).

Spectroscopy characteristics

To more investigate the functional groups in these products, we have also used XPS technique allowing the surface chemical binding. Figure 7 represent the survey analysis of studied products showing the main chemical element with their atomic orbital: C_{1s} and O_{1s} at binding energy 284.6 and 532 eV, respectively [10, 12, 16]. Table II shows all the obtained binding energies from XPS for these three carbonaceous products (graphene, MWNTs and biochar). For further a more detailed investigation, we able used the obtained deconvolutions of these inhomogeneous peaks for main chemical elements such as C1s and O1s as shown in figure 7 (below). They showed exactly the functional groups found in these products clarifying consequently the previous results.

For the chemical bonds at the C1s level, we observe in these carbon-based nanomaterials, whether in the amorphous (biochar) or in crystalline (MWNTs and graphene) carbonaceous form, the presence of five chemical bonds with different concentrations depending to product type, such as C= C (sp2) has a binding energy around 284.5 eV which is the strongest for the three products, accompanied with C-C (283 eV), C-O (285-286 eV), C=O (287 eV), O-CO (288-289 eV) and O-C=O (290 eV) [25].

Table. II
THE MAIN CHEMICAL ELEMENTS MAKING UP THESE STUDIED CARBON-BASED NANOMATERIALS.

Sample	Binding energy (eV)	Element	C1s (at.%)	O1s (at.%)
graphene	284.4	C1s	90	10
	532.5	O1s		
	978	$OKL_{23}L_{23}$		
	1225	$CKL_{23}L_{23}$		
MWNTs	284.6	C1s	87	6
	533.4	O1s		
	981	$OKL_{23}L_{23}$		
biochar	284.5	C1s	63	37
	530.5	O1s		
	978	$OKL_{23}L_{23}$		

Otherwise with regard to the O1s level, we indicate for MWNTs and graphene the dominant presence of O-C / C-O-C chemical bond level found around 532 eV with other secondary chemical bonds, like: O=C / O-C=O and the adsorbed water due to the storage effect.

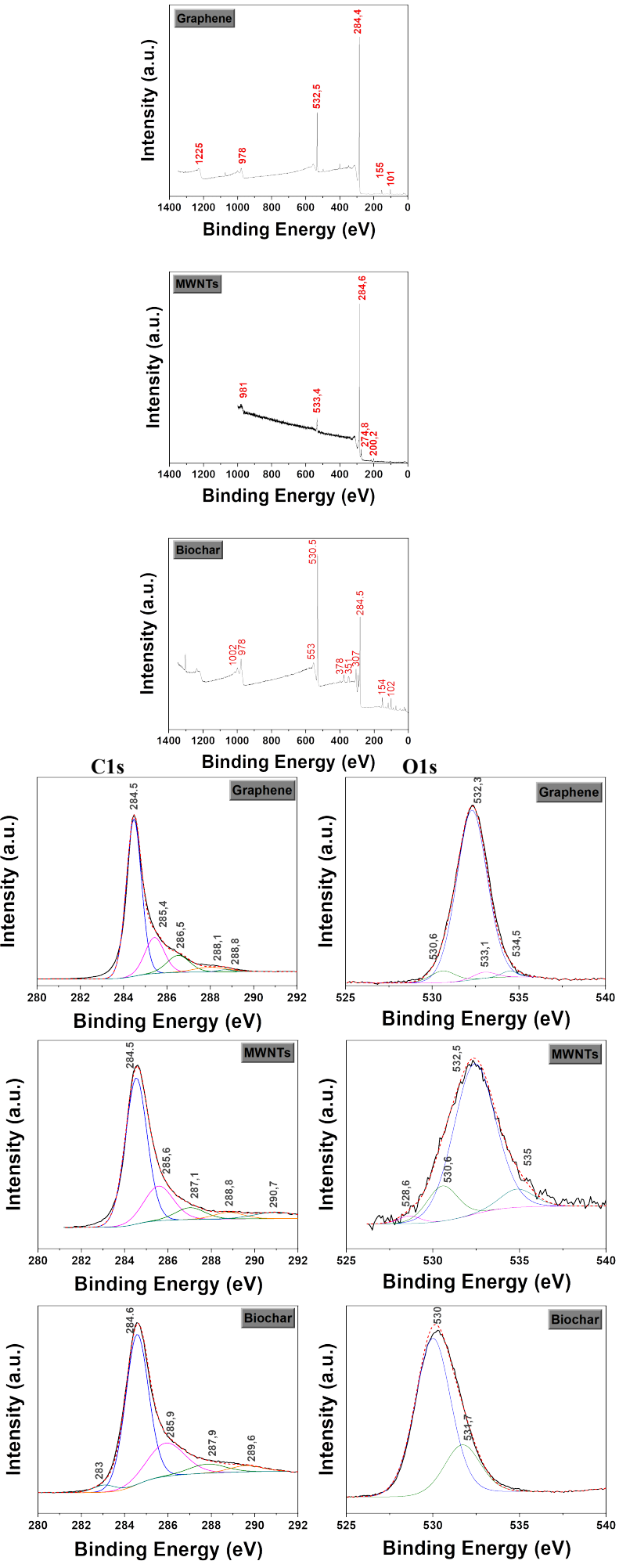

Fig. 7: Expanded XPS survey spectra and their deconvoluted C1s and O1s for graphene, MWNTs and biochar.

Whereas, in the case of biochar, the dominant chemical bond is found around 531 eV corresponding to the O=C or O-C=O bond accompanied just by another weak bond around 532 eV (O-C / C-O-C).

IV. Conclusion

According to these results that have been presented in this investigation after comparing physic-chemical properties of three kind of carbonaceous nanomaterial, MWNTs and graphene possess crystalline structures while biochar have amorphous structure with porous texture as proved precisely through their morphological, Raman and XPS spectroscopy. This difference focalizing on the porosity and specific surface area as well as their functional groups concentration and type is a main feature that makes each of them with particular application in various fields, like in: energy storage, environment depollution, biosensing and soil fertilization.

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