Chapter

Application of Titanium Dioxide in the Synthesis of Mesoporous Activated Carbon Derived from Agricultural Waste

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Abstract

Adsorption is an important technique that significances the characteristics of porous solid materials and fine powders. The importance of porous solid materials and fine powders has been recognized when porous coal used for various applications such as catalysis, separation, isolation, sensors, chromatography, etc. Herein, the synthesis of mesoporous activated carbon derived from agricultural waste using TiO_2. The TiO_2-modified carbon was characterized employing scanning electron microscope (SEM), attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopy, powder X-ray diffraction (pXRD), Brunauer–Emmett–Teller (BET) surface area analyzer and X-ray photoelectron spectroscopy (XPS). The obtained results suggested that the TiO_2-modified carbon could be a potential material for various application like dye removal, metal removal and allied areas. This book chapter describes the commonly used classifications of porous bulk materials and also reported here the characterization of porous solid materials and fine powders with special reference to the evaluation of the surface area, pore size distribution and thermodynamic parameters of the different mesoporous material, at various scales of resolution using relevant techniques. These materials comprise several levels of structures that of the mesopores, micropores as well as macropores. The apparent topography analysis of these materials, of various pore diameters, synthesized in our laboratory has been determined at various scales with the help of various characterization techniques.

Keywords: Titanium Dioxide, Mesoporous Materials, N_2 adsorption–desorption isotherm, Capillary (or pore) condensation

1. Introduction

Since ancient times, the importance of porous solid materials and fine powders has been recognized when porous coal used for its medicinal properties [1]. Research on porous solid materials and fine powders, which have various applications such as catalysis, separation, isolation, sensors, chromatography, etc., have reinforced the global interest [2] in environmental sustainability and energy storage. In the above - referenced applications, the outperformance of different porous solid materials and fine powders is extremely dependent on each material's internal porous structure. Therefore, the internal geometry, size, connectivity, etc. such as porous materials of different properties were fully characterized to better understand a particular physical process taking place in a porous medium [3]. Gas adsorption is one of many experimental methods available to characterize porous materials by the surface and pore size. These include ray and neutron scattering of small angles (SAXS and SANS), porosimetry of mercury, electron microscopy (scanning and transmission), thermoporometry, nuclear magnetic resonance methods, and others. Each method has a limited application scale for analyzing pore size. IUPAC recently provided an overview of different methods for characterizing pore size and their range of application [4]. Among these methods, gas adsorption is the most popular because it enables the evaluation of a wide range of pore sizes (from 0.35 nm to >100 nm), including the full range of micro-and mesopores and even macropores. Moreover, the techniques of gas adsorption are convenient to use and are not that cost-intensive compared to some of the other methods [5].

Adsorption finds very extensive potential application in the research laboratory and industry. Adsorption plays a very important role in various aspects of the catalysis of gases reactions by solid surfaces. As applications of adsorption from solutions may be mentioned the clarification of sugar liquors by charcoal, the removal of coloring materials from various types of solutions, and recovery of dyes from dilute solutions in several solvents. Adsorption has also been employed for the recovery and concentration of vitamins and other biological substances and finds immense utility in the chromatographic analysis [6]. The importance of the adsorption can be assessed in the sense that Langmuir was awarded Nobel Prize in Chemistry for his seminal contribution to surface science. Since then, two more Nobel Prizes Has been given in this area (Ertl in 1992 for his studies of chemical processes on solid surfaces and Marcus in 1992 for his theory and mechanism of electron transfer reactions on surfaces).

The materials comprising of pore size lying between Micro- and Macro-porous materials are regarded as mesoporous materials [7–9]. Mesoporous materials are the materials that have their pore size in between Micro- and Macro-porous materials. Meso a Greek prefix – "in-between" - micro and macroporous system. Following the IUPAC standard, microporous material has pore smaller than 2 nm while macroporous material possesses pores larger than 50 nm. Mesoporous material has high specific surface area due to high porosity within the mesopore range which forms the basis of their applications in varying fields. They possess high surface area -400-1000 m^2/g, large pore volume and high stability –500-600°C. A very common mesoporous material is customary activated carbon which is typically composed of a carbon framework having both mesoporosity and microporosity depending on the conditions under which it was manufactured. According to IUPAC, a mesoporous material can be deliberately ordered or disordered in a mesostructure [10–13]. In crystalline inorganic materials, mesoporous structure noticeably limits the number of lattice units, and this significantly changes the physics and chemistry. For example, the battery performance of mesoporous electroactive materials is significantly different from that of their bulk structure. "The overwhelming tendency for solids to minimize void space within their structure" is inherent, porous materials are difficult to make naturally. But Einstein says "In the middle of difficulty lies opportunity". The above statement was made true by the Mobil scientist in the year 1992 by successfully synthesizing the Mesoporous materials i.e. Mobil Crystalline Materials (MCM-41 and MCM-48) employing soft template strategy. This has opened new potentialities for Mesoporous materials, and extensive research has been contributed in this field.

Material can be classified as porous if its internal voids can be filled with gases. The history of porous materials began with the zeolites having an aluminosilicates framework which was synthesized employing a single template molecule with a small pore. Usually, they are synthesized by the use of the soft template method. Since then, research and development in this field have grown steadily. Notable examples of prospective industrial applications are catalysis, sorption, gas sensing, ion exchange, optics, and photovoltaics. Gas adsorption is the most effective and corroborative method for the characterization of the texture of porous solids and fine powders. The analysis was done on "Reporting Physisorption Data for Gas/Solid Systems" on the surface area and porosity [14, 15].

2. Materials and methods

The agriculture waste namely mustard cake (sample) was purchased from the local mill (Sai Enterprise, Lucknow, India). Sodium hydroxide (98%), hydrochloric acid (35.5%), potassium chloride (99.5%), sulfuric acid (98%) and TiO_2-Degussa P25 (ca.80% anatase, 20% rutile) chemicals were used and supplied by Bionic Enterprises, Lucknow, India. High-purity grade nitrogen gas (99.999%) and helium gas (99.9999%) were used for physicochemical characterization of the sample supplied by Krishna Gas Agencies, Lucknow, India. The double-distilled water (DDW) was prepared using a double distillation unit (Glassco Laboratory Equipment Pvt. Ltd., Ambala Cantt., India) and used for the preparation of modified material. All the reagents used for synthesis and experimental studies were of analytical and laboratory grade.

The agricultural waste sample was crushed and washed with double distilled water (DDW) and then sun-dried. This material was treated with 20 wt% H_2O_2 at 60°C for 24 h to oxidize the adhering organic matter and it was washed several times using DDW. This material was calcinated in the presence of O2 gas at 715°C for 25 min. The material, powdered activated carbon (PAC) was grounded and sieved to desired particle sizes. To get TiO_2-MMC, TiO_2-Degussa P25 (ca.80% anatase, 20% rutile) was added to PAC in a 5:1 weight ratio, in NaOH 2 N alkaline solution; in the 1000 ml, volumetric flask with a reflux condenser, the new potential material (TiO_2-MMC) was obtained from the slurry under stirring (300 ppm) for 24 h, at atmospheric pressure and 100°C, on a thermostat heating plate. After filtration, washing and drying at 105–120°C till a constant mass, the dried TiO_2-MMC was ground and passed through a 100 mesh sieve and stored in a desiccator. The material was characterized employing ATR- FTIR, SEM, XRD, BET and XPS [6, 7].

For the functional group analysis, the sample was directly (without any preparation) scanned in transmittance mode with a wave region ranging from 4000 to 500 cm^{-1} by ATR-FTIR (Bruker-Tensor 27) [16, 17]. The particle morphologies of samples were studied using SEM (JEOL JSM 6490LV). The sample was mounted on an aluminum stub with the help of double-sided tape. Mounted stubs were coated with gold–palladium for analysis using a Polaron sputter coater [16, 17]. The specific surface area, size and pore diameter of the materials were measured using a surface area analyzer (Belsorp-Mini-II, Japan). A definite amount of the adsorbent materials (0.20 g) were heated under pre-treatment at 120°C under vacuum for approximate 5–6 hrs. Subsequently, the process of N_2 adsorption and desorption was performed. The surface area and pore diameter were calculated using the standard Brunauer–Emmett–Teller (BET) equation and pore size was obtained employing a method of Barrett–Joyner–Halenda (BJH) and T-plot [16, 17]. The crystalline nature of the sample was examined by powder X-ray diffraction ($_P$XRD) (PAN analytical X'pert, Powder, Malvern Panalytical, UK) in 2-theta(degree) angle range 5°-80° at 45 kV using CuK radiation with 148.92 times per step [6, 7]. The XPS experiments were performed in an ultrahigh vacuum (UHV) using a high-resolution X-ray photoelectron spectrophotometer (HR-XPS), (PHI 5000 Versa Prob II). All samples were dehydrated under vacuum before XPS analysis. All Data of XPS analysis were plotted using Casa XPS Version 2.3.17PRI. I and Origin Pro 8 SRO v8.0724 (B724) software [16, 17].

3. Result and discussion

3.1 Functional group analysis

Vibrational spectroscopy in the infrared region was used to perform the characterization of the molecular structure of the adsorbent from the vibrational modes related to functional groups present in the sample. ATR FTIR spectroscopy provides evidence for specific functional groups on the surface of the TiO_2-modified mesoporous carbon (TiO_2-MMC). Several characteristics bands were observed in the ATR FTIR spectrum (**Figure 1**). Many existing groups in carbon derived from agriculture waste (CDAW) (blank) [18] are also present in the TiO_2-MMC, showing that modified material exhibits many of the same functional groups of CDAW (blank).

It was observed that the infrared spectra of TiO_2-MMC which overlapping with CDAW (source material) so that there are new peaks as observed in 3832.20–3425.4, 1632.21, 1570, 1046.50, 600.23, 500.30 cm^{-1}. The broad and strong bands at 3832.20–3425.4 cm^{-1} have been assigned because of bonded hydroxyl (-OH) or amine ($-NH_2$) groups. The band 1632.21 can be ascribed to –C=O stretching. The band around 1570 cm^{-1} can be ascribed to –C=C. The band at 1046.50 cm^{-1} is due to the C-O of carbonyl groups. The stretching band corresponding to Ti-O-Ti is clearly in the region of 509 cm^{-1} [18]. The band around 620.23 cm^{-1} is attributed to the Ti-O-C vibration which indicates that TiO_2 (Dugasa P25) were chemically bonded with carbonized carbon in TiO_2-MMC [16, 17].

3.2 Microstructural and elemental composition analysis

The SEM images of CDAW (**Figure 2a**) [16] and TiO_2-MMC are shown in **Figure 2b**, which demonstrate the porosity and texture of TiO_2-MMC. It has been observed that TiO_2-MMC retain the surface characteristics of its precursor (CDAW and TiO_2)

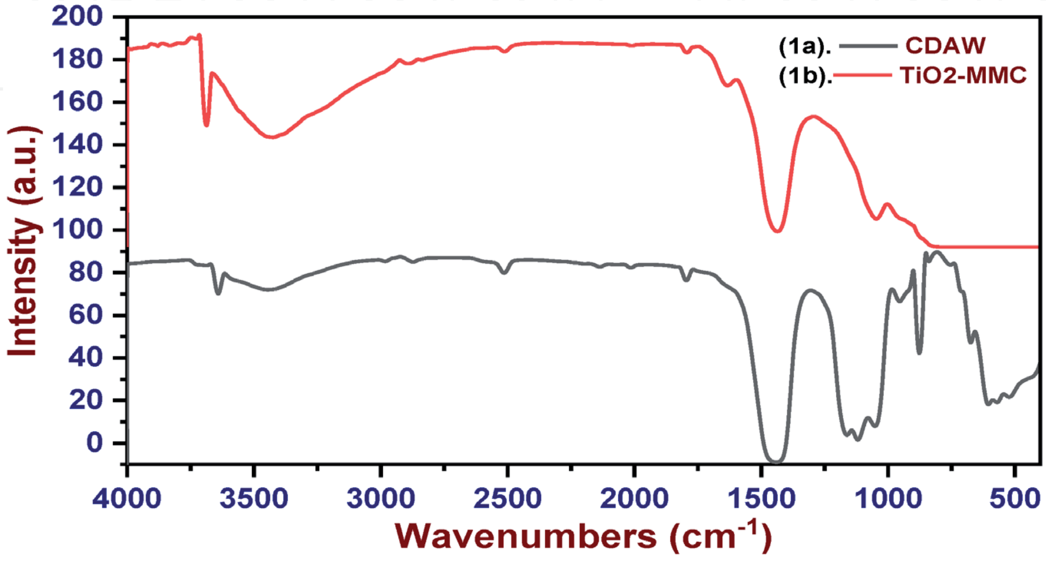

Figure 1.
FTIR spectra of (1a). CDAW and (1b) TiO2-MMC.

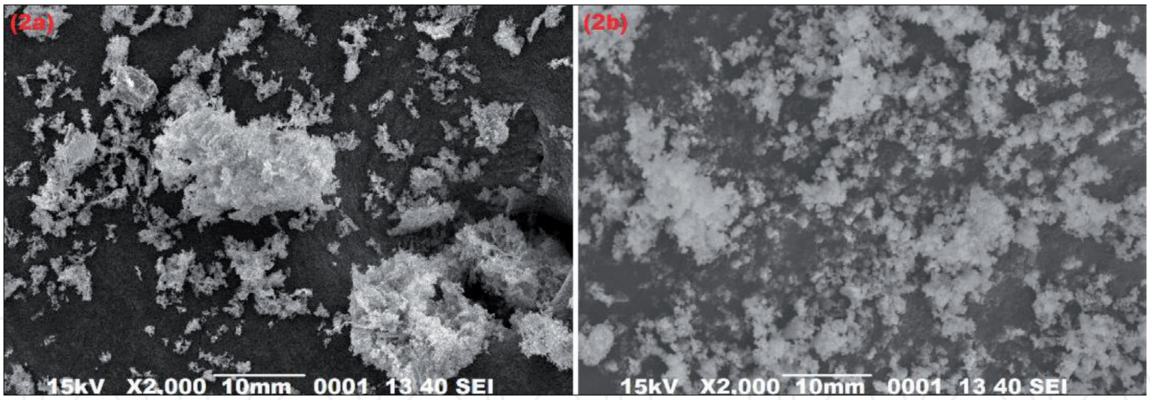

Figure 2.
SEM micrographs of (2a). CDAW (blank) and (2b) TiO2-MMC.

as shown in **Figure 2**. However, with some remarkable modifications, the results (**Figure 2b**) obtained showed that there was displacement as the temperature of thermal decomposition of the modified adsorbent suggesting that the modification caused a difference in structure. The SEM micrographs (**Figure 2b**) show a spongy aspect, fibrous surface with irregular and heterogeneous structure with many pits and fissures through the surface prominent possibly due to powerful oxidizing agent H_2O_2, which caused some changes in the surface of TiO_2-MMC.

3.3 Specific surface area, particle size measurement

The most applicable and accessed procedure for evaluating the surface area of porous and finely-divided material is the Brunauer–Emmett–Teller method [6]. Given the specific controlled conditions, the BET-area of a nonporous, macroporous or mesoporous solid can be referred to as the ‘probe accessible area’ (which is the

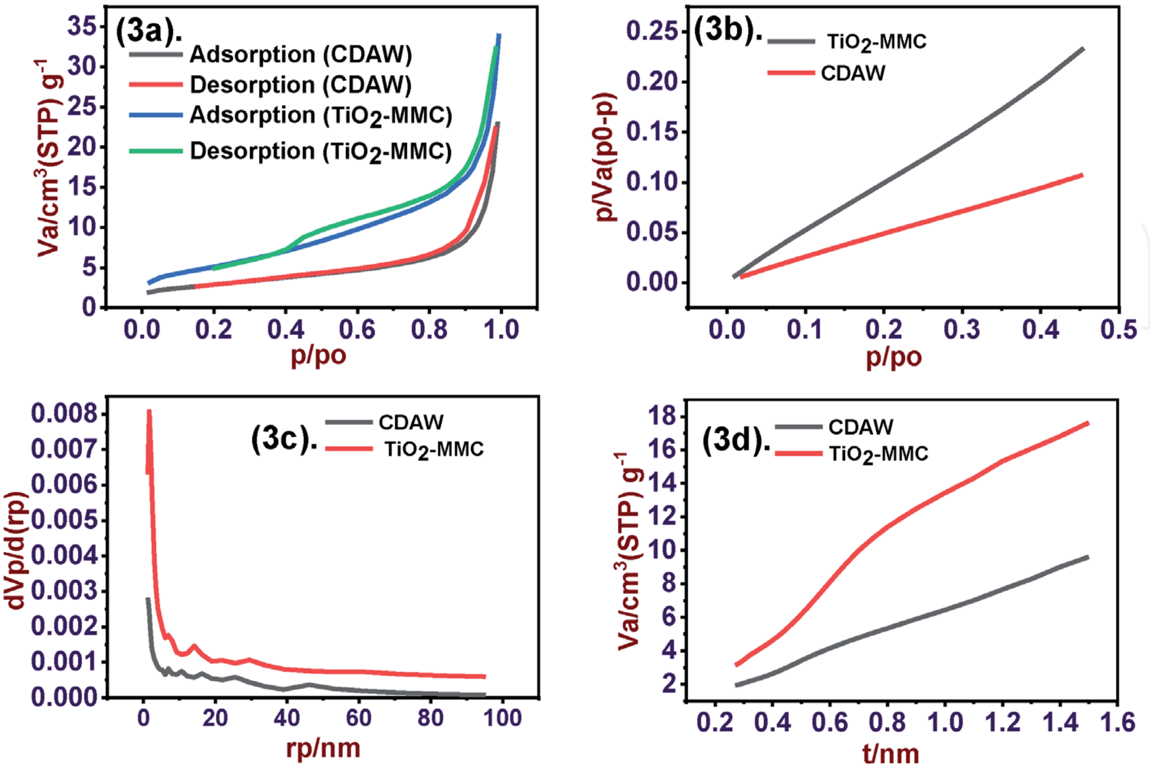

Figure 3.
BET analysis for CDAW and TiO2-MMC, (3a). N2 adsorption and desorption, (3b). BET, (3c). BJH and (3d). T-plot respectively.

effective area available for the adsorption of the specified adsorptive). There are two particular stages involved in the application of the BET method. It is important to transform a physisorption isotherm into the 'BET plot'. The calculated value of the BET area is dependent on (i). The adsorptive and operational temperature and (ii). The procedure used to locate the pressure range in applying the BET equation.

The N_2 adsorption–desorption of both CDAW and TiO_2-MMC was conducted (**Figure 3a**). BET analysis (**Figure 3**) confirms that TiO_2-MMC falls on the mesoporous domain. The typical specific surface area of TiO_2-MMC was found as S_{BET} = 18.845 m^2g^{-1} whereas the surface area of the CDAW was typically S_{BET} = 10.251 m^2g^{-1} (**Figure 3b**). In the case of fine and especially, free-floating powdered samples can present an additional difficulty to the during BET analysis. Particles may also be deposited on valve seats causing problematical leaks. This loss of powder from the confinement of the sample bulb, known as elutriation, should be controlled and eliminated as far as is reasonably practicable.

The adsorption/desorption isotherm (**Figure 3a**) shows the relationship between the amount of adsorbed/desorbed gas (y-axis) and the pressure of adsorptive (x-axis) at the constant temperature. Nitrogen (at the boiling temperature of 77 K) was the conventional and usual choice for the adsorptive to obtain (BET), with $\sigma m(N_2)$ assumed to be 0.162 nm^2 and being a close-packed monolayer. Moreover, liquid nitrogen was readily available and also nitrogen isotherms on many adsorbents were found to exhibit a well-defined Point B. Though, present studies have shown that due to its quadrupole moment, the orientation of a nitrogen molecule is dependent on the surface chemistry of the adsorbent. And, this could lead to the tentative reading of the value of σm (N_2) – possibly ~ 20% for some surfaces [7].

A more suitable alternative adsorptive for surface area analysis could be Argon. Argon does not have a quadrupole moment and is less reactive than the diatomic nitrogen molecule. Argon, though, at 77 K is, possibly, less reliable than Nitrogen. Argon adsorption at 87 K (liquid argon temperature) is the possible alternative. At 87 K, a cross-sectional area, $\sigma m(Ar)$, of 0.142 nm^2 is assumed. Due to the absence of a quadrupole moment and the higher temperature, σm (Ar) is less prone to the differences in the structure of the adsorbent surface. Argon adsorption at 87 K facilitates the micropore analysis. Analysis at 87 K can be done by using either liquid argon (replacing liquid nitrogen) or a cryostat (or cryocooler) [1, 7]. Manometric adsorption equipment of maximum accuracy can effectively assess the surface areas as low as (~ 0.5–1) m^2 with nitrogen or argon as the adsorptive. For lower surface areas, krypton adsorption at 77 K is the preferred adsorptive. Though krypton at 77 K is similar to argon at 77 K. Hence, the standard thermodynamic state of the adsorbed layer is not accurately gauged.

These methods (**Figure 3**) used to calculate the micropore radius (cylindrical shape), micropore diameter (cylindrical shape), area distribution, volume distribution, Integral curve, pore specific surface area and pore volume. Methods for mesopore size analysis have been proposed by Barrett, Joyner, and Hlenda (BJH) (**Figure 3c**) [19, 20]. The preadsorbed multilayer film can be accounted for with the integration of the Kelvin equation with a standard isotherm (the t-curve) to analyze the specific nonporous solids. Still, for analyzing the accurate size of narrow mesopores, the standard t-curve is not a convincing procedure (**Figure 3d**), the validity of the Kelvin equation also provides an approximation only because the mesopore width is reduced in the procedure. Studies have proven that the Kelvin equation-based procedures, such as the BJH method, underestimate the pore size for narrow mesopores.

The IUPAC classification, where pores are classified in macro-, meso-, and micropores, is mostly based on the different mechanisms that occur in these pores

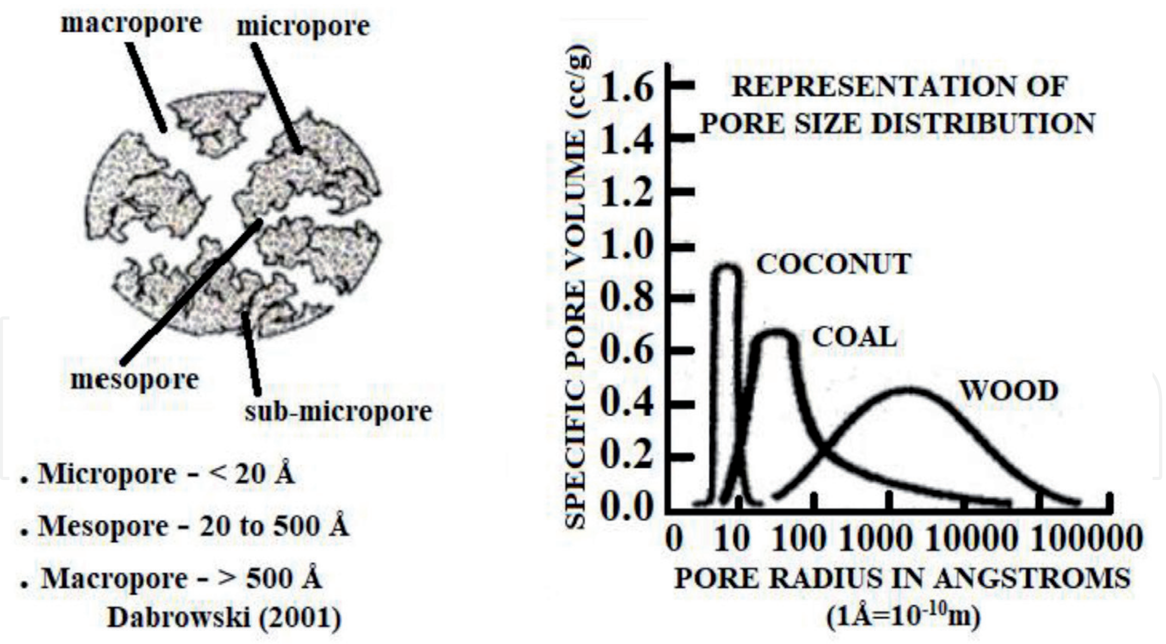

Figure 4.
IUPAC classification of pores. Figure adapted from reference [1]. © 2015 IUPAC & De Gruyter.

during 77 K and 1 atm isothermal adsorption of N_2 Pressure (**Figure 4**). Multilayer adsorption, capillary condensation, and microporous filling, respectively, are processes related to macropores, mesopores, and micropores. Depending on the different relative pressures p/p° ratio, the pore width classes correspond to the application of the capillary condensation theory. The 50 nm pore width is associated with 0.96 relative pressures. Above this value, isothermic adsorption experiments are quite difficult to interpret and the capillary condensation theory of applicability has not been adequately tested [21–25].

3.4 Phase determination

The pXRD spectra shown in **Figure 5** illustrates that the treated CDAW and modified with TiO_2 under optimum preparation condition is crystalline in structure. There are seven broad peaks centred on a 2θ value of 250, 300, 370, 470, 520, 540 and 630. $2\theta = 68°$ is not clearly described [21–25].

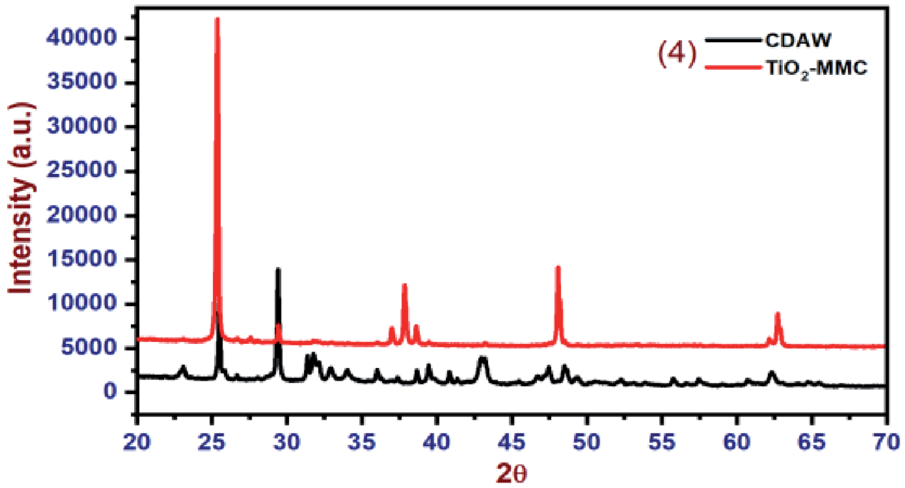

Figure 5.
XRD spectra of CDAW and TiO2-MMC.

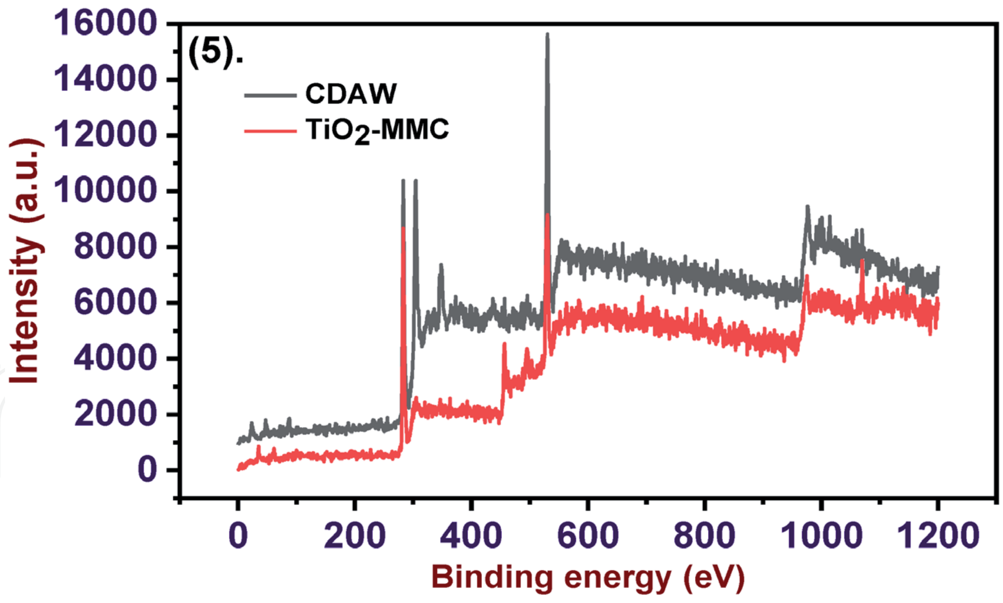

Figure 6.
XPS survey AlKα PES of CDAW and spectra CDAW.

3.5 XPS analysis

To determine the chemical composition of the CDAW and TiO_2-MMC XPS analysis was performed. The surveys scan of CDAW in (**Figure 6**) illustrated that its oxygen content is around 17.4%, suggesting the containing of abundant surface oxygenic functional groups. In the C 1 s XPS spectra of CDAW (**Figure 6**), the peaks at 284.8, 286.7, and 288.9 eV are ascribed to the sp2 hybridized (C–C), alcoholic (C–OH) and carbonyl (C=O) carbon atoms, respectively.

The XPS survey scans of TiO_2-MMC (**Figure 6**) show the typical spectra of TiO_2-MMC (P25), and the more intense feature peak of C1s in TiO_2-MMC was attributed to the layer of the hybrid. Concerning the C1s XPS spectra of TiO_2-MMC, aside from the above listed characteristic peaks, it was noticed that an additional peak emerges at the binding energy of 283.5 eV, which is higher than that of titanium carbides ($Ti4^+$–C) and should be assigned to the carbon bonded with oxygen-deficient titanium ion ($Ti3^+$–C) revealing the distinctive chemically bonded carbon/TiO_2-MMC. Besides, the atomic ratios of O: C for PAMC and TiO_2-MMC are 0.21 and 0.17, respectively, suggesting the decreased oxygen content of coated PAMC TiO_2-MMC [21–25].

4. Conclusions

- This chapter briefly introduced the synthesis of impregnated titanium dioxide mesoporous carbon derived from agricultural waste and high-resolution characterization techniques and reliable commercial instrumentation.

- The AT-FTIR analysis revealed the specific functional groups present in the TiO_2-MMC. The microstructural analysis confirmed the chemical composition and surface nature.

- The initial IUPAC classifications of physisorption isotherms and hysteresis loops have encapsulated the recent characteristic types the Brunauer–Emmett–Teller (BET) method for the analysis of surface area needs careful attention.

- The nitrogen adsorption isotherms, pores size distribution and scanning electron micrographs of TiO_2-MMC suggest that it could be very good mesoporous materials for various applications.

- Relying on the standard methods for the analysis of the surface area and pore size can lead to inconclusive BET surface areas and pore size distributions. Such gaps have necessitated the introduction of more convincing and validated pore models based on the non-rigid nature of the adsorbent.

- The study highlights that the pore size analysis of narrow mesopores remains inconclusive by enlisting the procedures derived from the Kelvin equation, as the Barrett–Joyner–Halenda (BJH) method.

- The classification of non-rigid adsorbents poses another area for further investigations. Enhanced procedures for standard analysis and development of the latest certified reference materials are the imminent need of research in this domain.

Nomenclature

SEM	Scanning Electron Microscope
ATR-FTIR	Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy
$_P$XRD	Powder X-ray Diffraction
BET	Brunauer–Emmett–Teller
XPS	X-ray Photoelectron Spectroscopy
SANS	Small-Angle Neutron Scattering
SAXS	Small-Angle X-ray Scattering
IUPAC	International Union of Pure and Applied Chemistry
MCM	Mobil Crystalline Materials
PAC	Powdered Activated Carbon
TiO_2-MMC	Titanium Dioxide-Modified Mesoporous Carbon
BJH	Barrett–Joyner–Halenda Method
S_{BET}	Specific Surface Area
Ultrahigh Vacuum	UHV
HR-XPS	High-resolution X-ray Photoelectron Spectrophotometer
CDAW	Carbon Derived from Agriculture Waste

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