Preparation, Characterization and studies with Diphenyl ether (DPE) Decorated Nanomaterials

A thesis submitted in the partial fulfilment
of the requirement for the degree of

MASTER OF SCIENCE
IN
CHEMISTRY
by
Deepika
(301602014)

THAPAR INSTITUTE
OF ENGINEERING & TECHNOLOGY
(Deemed to be University)

Under the supervision of

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JUNE 2018

Candidate's Declaration

I hereby declare that work being presented in this dissertation entitled "**Preparation, Characterization and studies with Diphenyl ether (DPE) Decorated Nanomaterials**" in partial fulfilment of the requirement for the award of **Master of Science in Chemistry** carried out at School of Chemistry and Biochemistry, Thapar Institute of Engineering and Technology, Patiala is my own carried out under the supervision of **Dr. Manmohan Chhibber** and **Dr. Loveleen Kaur Brar** TIET, Patiala during the period **January to June 2018**.

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Place : Patiala
Date : 15th June 2018

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This is to certify that the statement made above by the candidate is correct and true to the best of my knowledge.

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Certificate

This is to certify that the dissertation entitled "**Preparation, Characterization and studies with Diphenyl ether (DPE) Decorated Nanomaterials** " being submitted by **Deepika (Roll No 301602014)** in partial fulfilment of the requirement for the award of degree of **Master of Science in Chemistry** at School of Chemistry and Biochemistry, Thapar Institute of Engineering and Technology, Patiala is an bona fide work carried out under the supervision of **Dr. Manmohan Chhibber** and **Dr. Loveleen Kaur Brar** TIET, Patiala and that no part of this thesis has been submitted for the award of any degree.

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Deepika

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Dedicated to my Family

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ABSTRACT

Carbon nanospheres (CNS) find applications in many areas of science like batteries, adsorption of metal ions and organic dyes and many more. Due to presence of dangling functional groups on its surface, including free hydroxy group, there is a possibility to tag these materials with useful organic compounds that will enhance its application in different fields. For example, compounds with antibacterial properties can be tagged on to the surface of CNS to create antibacterial surfaces that can be very useful in the development of new materials that will find applications in surgery and hospitals. This work shows the tagging of a diphenyl ether on the surface of CNS by two methods. Product obtained using both the methods were characterized using UV-Vis spectroscopy, Fourier Transformation Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). Results indicated change in the morphology of the CNS after tagging at lower concentrations of CNS and diphenyl ether. Corresponding results were corroborated with FTIR and UV-Vis spectroscopy also.

INTRODUCTION AND LITERATURE REVIEW

Michael Faraday in 1857 reported the formation of gold solution that when illuminated, reflects a golden character due to its minuteness. Diffused gold, as it was then termed, in the solution was reported to be present in abundance compared to quantity of the solid gold [1]. A century later, after the investigations on electron microscope, average diameter of diffused gold was determined to be (6±2) nm [2]. Later, after the discovery of high-resolution electron microscope the range of these diffused particles was found to be between 3 to 30 nm [3]. Thus, the era outlined above, formed detrimental years of the world that was to explode a century and half later to create a new branch of science called nanoscience.

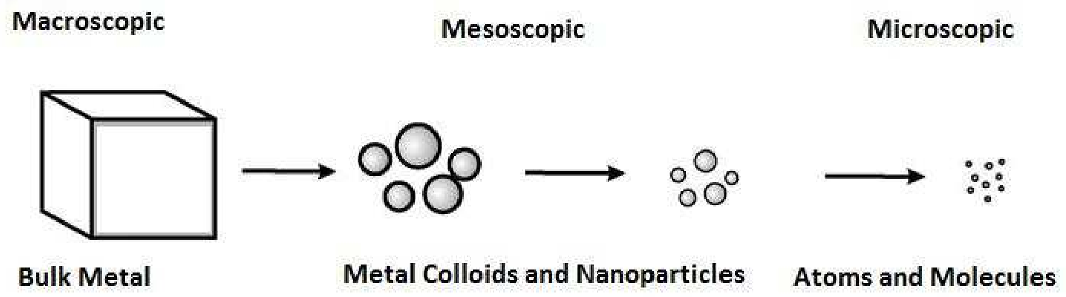


Figure –1: Different organization of matter obtained after fragmentation of bulk metal to mesoscopic and to microscopic levels.

Edwards et. al. described the mechanism for the formation of microscopic particles from the macroscopic particles **(Figure –1)**[4]. The explosive development of gold nanoparticles included their application in many fields. For example, Haruta et al., reported that nanoparticles of gold of 2-4 nm diameter, if dispersed on metal oxides such as Fe_2O_3 and NiO, could catalyze the conversion of poisonous carbon monoxide gas into its constituents even at subzero temperatures[5]. The discovery, if implemented, could result in tremendous load reduction of carbon monoxide, due to vehicular and other emissions, on earth. Later, same and many other interrelated phenomena were observed with native gold nanoparticles[6,7].

Although gold nanoparticles were the first to be observed and find use, applications of other metal nanoparticles also revolutionized world. Zinc oxide, which is an essential ingredient of many enzymes and other biological processes, when converted to corresponding nanoparticles could be used in sun screens due to former's wide bandgap[8]. Similarly, these very nanoparticles get activated by light to diffuse into the bacterial cell wall for their application as antimicrobials[9]. It was also observed that antimicrobial activity increased with increase in surface-to-volume ratio due to a decrease in the particle size of zinc oxide nanoparticles[10] as shown in **Figure 2**.

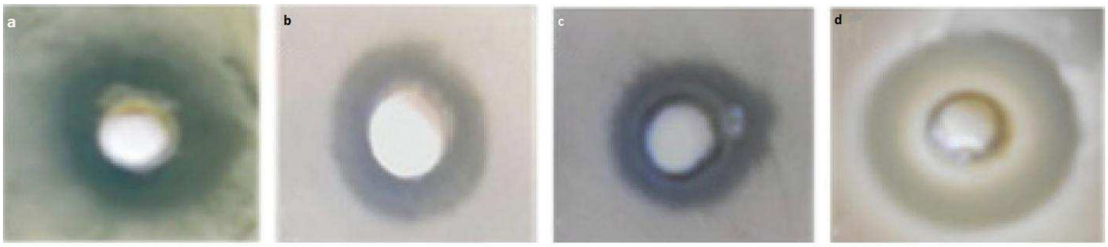


Figure-2: Zone of inhibition created by ZnO nanoparticles against a) Escherichia coli, b) *Staphylococcus aureus*, c) *Pseudomonas aeruginosa*, and d) *Bacillus subtilis.*

Magnetic iron oxide (Fe_3O_4 and γ-Fe_2O_3) nanoparticles have low toxicity, super-paramagnetic properties and high surface to volume ratio. These advantages along with the advantage of simple synthesis and the separation allow their use in diagnostics like magnetic resonance imaging (MRI), thermal therapy, and drug delivery [11]. Gupta et. al. in a review have extensively discussed the surface modification of magnetic nanoparticles and its effect on the stability and magnetization. Their biomedical applications have also been discussed[12]. **Figure-3** below shows TEM images nanoparticles prepared by two different techniques.

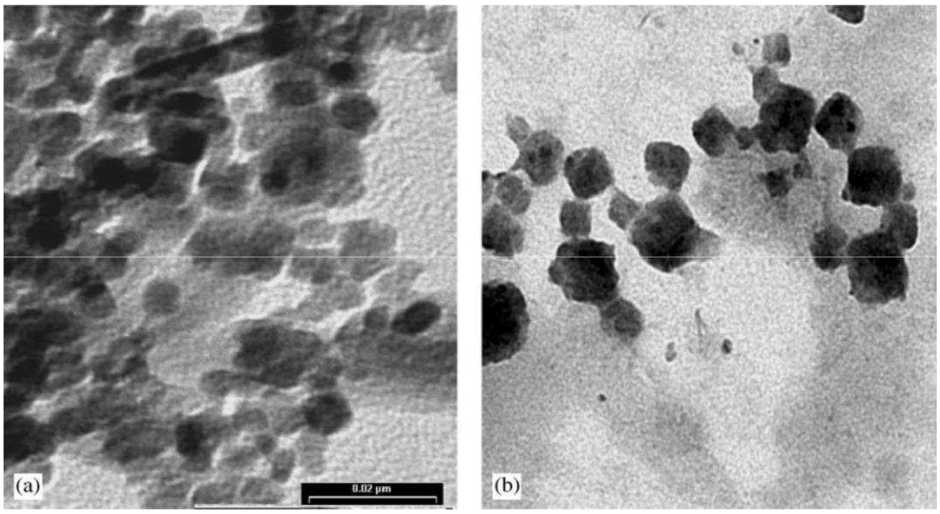


Figure- 3: TEM images of magnetic nanoparticles prepared by **(a)** bulk solutions **(b)** reverse micelle method

Besides gold, silver, iron and zinc nanoparticles, silica was also one of the earliest materials that found applications in many industrial fields such as additives in the drugs, cosmetics, printing toners and various packaging materials. However, their potential toxicity is still a matter of concern for many applications. Pompa's group from Italy carried out systematic in vitro experiments to study biological impact of SiO_2 nanoparticles (**Figure- 4**) of three different sizes (115, 60 and 25 nm) with as many as five cell lines. No detectable cyctotoxic effects in all the cell lines were observed up to a concentration of 2.5 nM. Beyond that, uptake by the cell was dependent upon size but was independent of the charge[13].

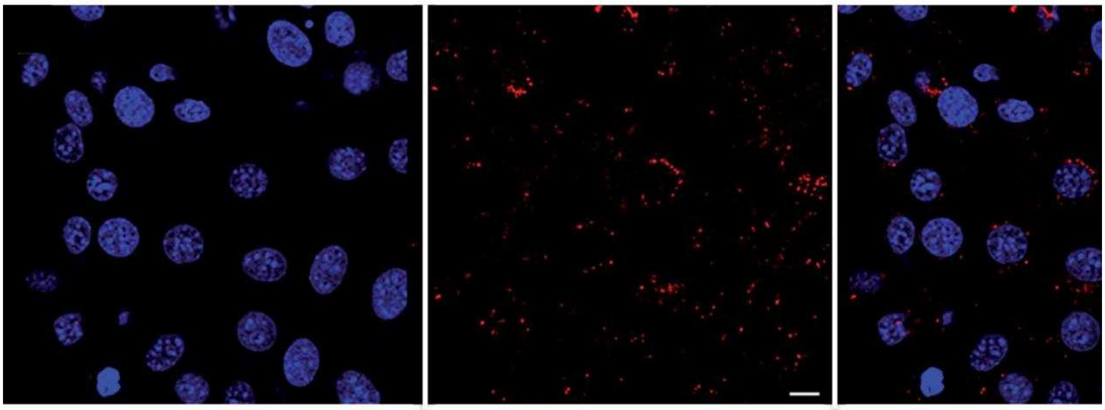


Figure - 4: Confocal images of cells treated with quantum dots doped SiO_2 NPs of 25nm (10nM) for 48 h **Left:** Nuclear staining; **Center:** QDs fluorescence; **Right:** merged image (Scale bars: 10 mm)

One of the important land marks in the nanoscience was the discovery of fullerenes. In fact, in the carbon-based nanomaterials, bucky balls - as the fullerenes are usually named, take the first place. Kroto, Curl and Smalley were awarded 1996 Nobel Prize in chemistry for this discovery that had potential applications. Earlier attempts to convert this hydrophobic, highly conjugated electron deficient substance into water soluble material resulted into diverse molecules that have applications as antiviral compounds, photosensitizes, antioxidants, drug and gene delivery materials besides many diagnostic applications[14]. In fact, the discovery of fullerenes led to the discovery of carbon nanotubes when Iijima, in 1991, while working with fullerene shoot reported a new material, now known as nanotubes, that had layers of concentric tubes (in micro scale). The discovery revolutionized the fields of electronics and preparation of mechanical devices due to its strength and many other properties like easy emission of electrons, sensors and capability to store gaseous substances[15]. This was followed by discovery of honeycomb sp^2 carbon lattice of one atom thickness named graphene[16]. Graphite, although being known for more than five centuries has layered structure, Geim *et. al* discovered graphene sheets [17] that further led to discovery of graphene flakes, nanoribbons[18], and controlled (three dimensional, two dimensional and single dimensional) graphene sheets [19]. Of late this material due to its unique properties has [16, 18] wide utility in ultra-capacitors[16,] transistors, field emission sensors (gas and bio), transparent electrodes and in lithium- ion batteries[20]. **Figure -5** below shows the evolution of carbon-based nanomaterials from fullerenes to graphene.

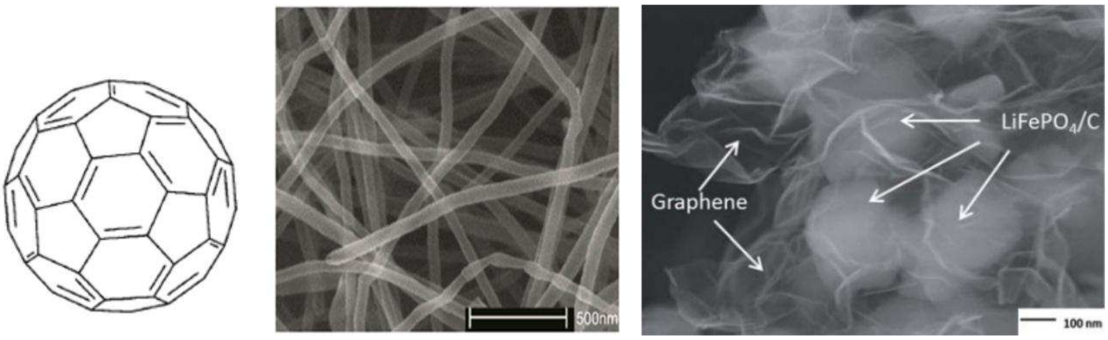


Figure -5: Evolution of carbon-based nanomaterials **Left:** Unique structure of fullerene **Center:** SEM image of CNT[21] **Right:** Single layered sheet of graphene as commercially available today.

The tendency of carbon to form carbon-carbon double bond network is the central theme that forms molecular basis for all the above mentioned three types of materials and carbon nanotechnology. These materials originally or by tweaking their molecular rearrangement can form very diverse materials that have features like light-weightiness, variability of the structures (like tubes, onions, cones etc.), conducting properties (conducting, insulating as well as intermediary properties) and thermal conductivity[16].

Carbon nanospheres (CNS) are another class of useful nanomaterials that have been reported in many applications recently. These nanospheres result from the pairing of carbon rings having pentagonal and heptagonal shapes[22]. Depending upon the conditions of the synthesis CNS can have sizes ranging from 50nM to 1µM. **Figure- 6** below shows SEM and TEM images of carbon nanospheres where they are present as agglomerated spherical bodies, rather than discrete entities due to high surface activity as a result of dangling bonds on to their surface. Actually, CNS were synthesized accidently by two major routes. Hence their fabrication can be carried out either by decomposition of carbon at high temperatures or pyrolysis at low temperatures.

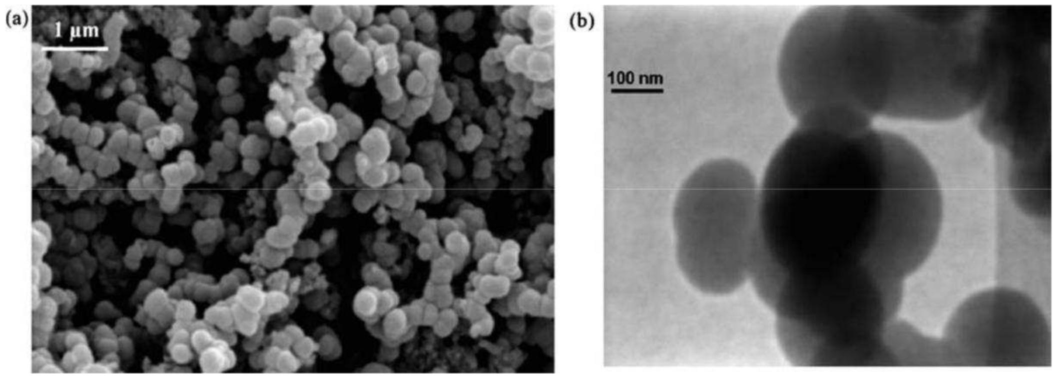


Figure – 6: **(a)** SEM and **(b)** TEM images of carbon nano spheres

Methods described below are some of the prevailing ones for the synthesis of CNS:

(i) **Arc- discharge process** involves use of high temperature generated by discharge arc, carbon electrode (negative) that itself sublimes to provide solid CNS at the positive electrode.

(ii) **Laser vapourization and plasma progression** uses vaporization of a carbon source in the presence of a metal catalyst using heated plasma using laser pulsation to vapourize the target. CNS, in this process can be obtained as per requirement using the choice of electrode.

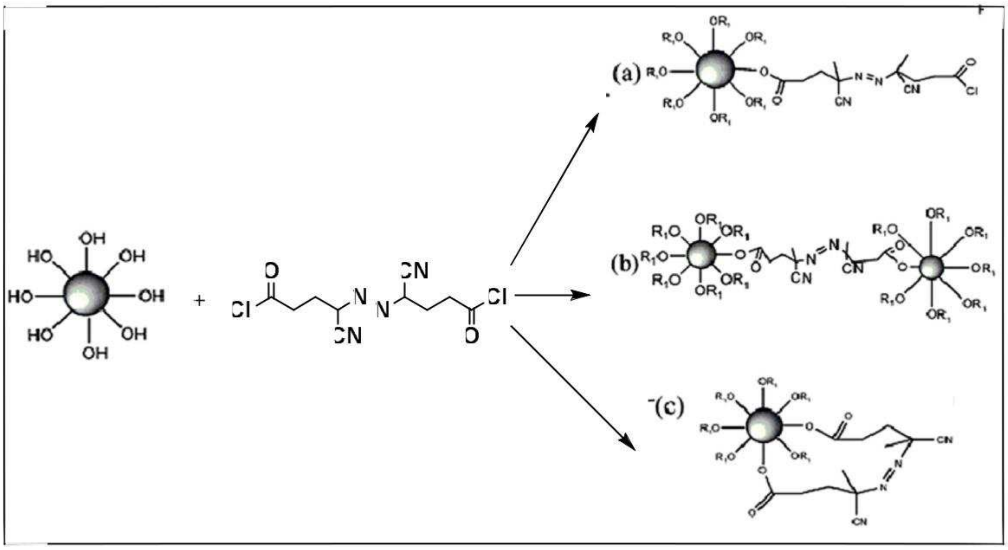
(iii) **Shock compression method** uses a carbon source at high temperatures, mostly the fullerene powder, to grow CNS.

(iv) **Chemical vapor deposition:** A volatile carbon precursor gets changed into a solid carbon. Out of many sub- categories, injecting the initial chemicals is direct.

(v) **Methods involving autoclaves** are very useful in controlling the size of CNS. Reactants**,** any carbon compound, are subjected to high pressure in an autoclave at variable thermal conditions to CNS. The concentration of carbon precursor can be used to fully control the size of material.

(vi) **Carbonization** is used as a carbon precursor in the solution form to forms CNS [23]

Many researchers have carried out surface functionalization of CNS with an objective to increase its charge holding capacity, enhance water solubility and many sensor applications. This includes both electrochemical and organic methodologies. One of the organic methodologies that included enhancing charge holding capacity has been shown in **Scheme-1** below.



Scheme-1: Part of the scheme to carry out surface functionalization of CNS with an objective to increase its charge holding capacity.

We envisaged carrying out surface functionalization of CNS with an objective to tag with organic molecule that has antibacterial and ion sensing properties. Aryl ether molecules are known to be antibacterial due to their ability to inhibit fatty acid biosynthesis. Starting with triclosan **(Figure -7)** a number of them have been synthesized by various research groups. Aryl ethers are also known to interact with Transthyretin (TTR), a protein susceptible to fibrillogenesis, preventing the cause of pathogenisis[24]. Chhibber's group has shown the use of diphenyl ethers for selective detection of some ions[25,26].

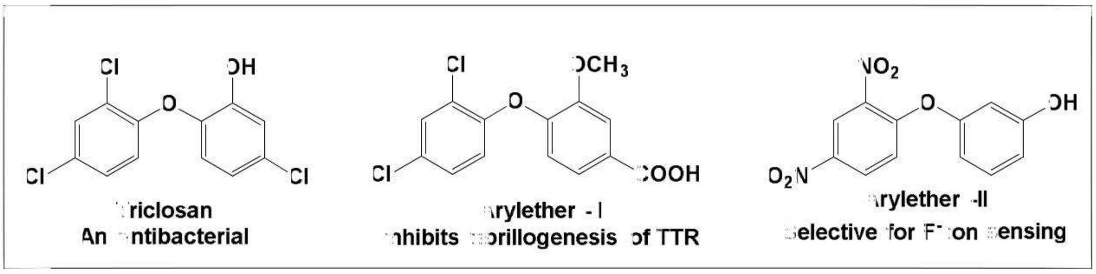


Figure -7: Diaryl ether molecules having different applications. Triclosan is a known antibacterial, Aryl ether-I inhibits the fibrillogenesis of TTR and aryl ether –II selectively binds to F^- anion in acetonitrile medium.

Following sections present the work that has been carried out to tag a diphenyl ether molecule on the surface of CNS. The synthesis and characterization of a diphenyl ether was carried out and tagging was done using organic synthesis on the already synthesized CNS of fixed size.

MATERIALS AND METHODS

General: All chemicals and solvents were procured from M/S AVRA chemicals, Hyderabad, India, of LR grade. Purification of organic compounds was done by column chromatography using silica gel (60-120 mesh) and specified solvent. TLC monitoring was done using silica coated aluminium sheets (60 F_{254}; 0.2mm thickness, Merck, India). Carbon Nanospheres(CNS) of dimensions 470 nm were a kind gift from Dr. Brar, SPMS, TIET, Patiala.

Instrumentation: Characterization of organic compounds and synthesized materials was done using 1H (400 MHz) and 13 C NMR spectrometer (JEOL, model A1, Sai Labs, Patiala, India), Scanning electron microscope, SEM (JEOL, JSM, 6510LV, SAI Labs, Patiala, India). Infra-red spectrophotometer having attenuated total reflection facility (Bruker alpha, IISER, Mohali, India) and UV -Vis spectrophotometer (Hitachi, U-3900H, SPMS, TIET, Patiala, India). The centrifuge machine (Thermofisher Scientific), sonicator (Branson 3510) and rotatory evaporator (Roteva) was used for various processes.

METHODOLOGY

Synthesis of 3-methoxy-4-(2-nitrophenoxy) benzaldehyde (DPE-I): The synthesis was done by a known procedure[27]**.** Vanillin (500mg, 3.2mmol) and o-fluoronitrobenzene (463mg, 3.2mmol) were taken in a round bottom flask and dissolved in dimethylformamide (DMF). K_2CO_3(545mg, 3.9mmol) and 18-crown-6 (catalytic amount) were added and allowed to stir for 12 hours. Several batches with same amount were made. After completion of the reaction (TLC monitoring), work-up was done using dichloromethane (DCM) and water. The DCM layer was collected and again washed with alkaline water (aq. NaOH diluted) to remove traces of vanillin by making its sodium salt. The combined organic layer was washed with dil. HCl (25ml) to make it neutral (to pH paper) and water (4×100ml). The crude product was purified by column chromatography using solvents hexane (90%) and ethyl acetate (10%) to get pure product having yield 378mg (75.6%). ^{1}H and ^{13}C NMR was done to confirm the purity. ^{1}H NMR (400MHz, $CDCl_3$): δ 3.91(s,3H); 7.00(dd, J= 8.24 Hz, 1H); 7.05(d, J= 8.24 Hz, 1H); 7.28(dd, J= 7.32 Hz, 1H); 7.45(dd, J= 8.26Hz, 1H); 7.49(m,2H); 8.03(dd, J=8.26Hz,1H); 9.9(s, 1H). ^{13}C NMR ($CDCl_3$): 56.21, 111.15, 119.33, 120.54, 124.06, 125.43, 125.98, 133.63, 134.43, 140.91, 149.69, 149.85, 151.18, 190.80.

Synthesis of DPE -II: DPE-I synthesized above (500mg, 1.83mmol) was heated (65-70℃) and stirred in water (10 ml) and potassium permanganate solution (434mg, 2.74mmol) was added dropwise from top. The reaction mixture was heated for 2 hours and completion of reaction was checked (TLC monitoring- **Figure 8**). MnO_2 formed was removed by filtration and HCl (pH=2) was added to the aqueous layer to get white coloured solid. The product obtained was dissolved in ethylacetate and washed with water. The solvent was evaporated to get crude product that was purified using column chromatography (10% toluene and 90% methanol) having yield 0.184gm (36.8%). Confirmation of the product was done by NMR (^1H and ^{13}C). ^1H NMR (400MHz, DMSO): δ 3.70(s, 3H); 6.78(d, J=8.26, 1H); 7.03(d, J= 8.24 Hz, 1H); 7.20(m, 1H); 7.55(m, 2H); 7.69(s, 1H); 7.98(dd, j= 8.02 Hz, 1H).^{13}C NMR (DMSO): 55.62, 117.53, 120.37, 122.32, 122.62, 125.45, 134.80, 139.55, 143.15, 149.71, 150.60, 169.52, 169.63.

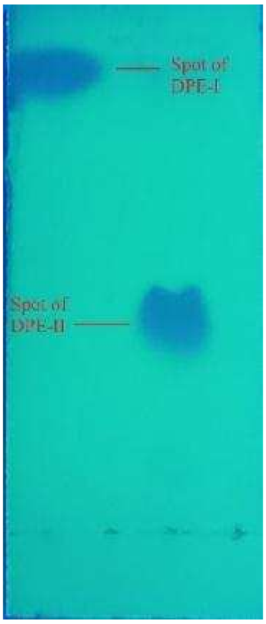


Figure - 8: Completion of oxidation reaction and formation of DPE-II from DPE-I

Surface functionalization of Carbon Nanospheres (CNS):

a) **Synthesis of DPE-III-A:** Sucrose generated nanospheres (3 ml (0.028M), 0.08 mmol), DPE-II (50 mg, 0.17 mmol) and conc. H_2SO_4 were taken together in a round bottom flask and refluxed for 5 hours at 60-70℃ in an oil bath.

b) **Synthesis of DPE-III-B:** DPE-II (50mg, 0.17 mmol) was taken in a round bottom flask and PCl_3. was added dropwise. The reaction mixture was heated in oil bath without any solvent till the evolution of HCl was ceased. The pungent smelling compound was used as such after dissolving in acetone. To a sonicated solution of nanospheres (3ml (0.028M), 0.08 mmol) and K_2CO_3 in acetone and water (1:1) mixture (10 ml), was added dropwise.

After 15 minutes, the reaction mixture was centrifuged at 8000 rpm (10℃) and washed with ethanol (4× 50 ml) and water (4×50ml) to remove any organic compound.

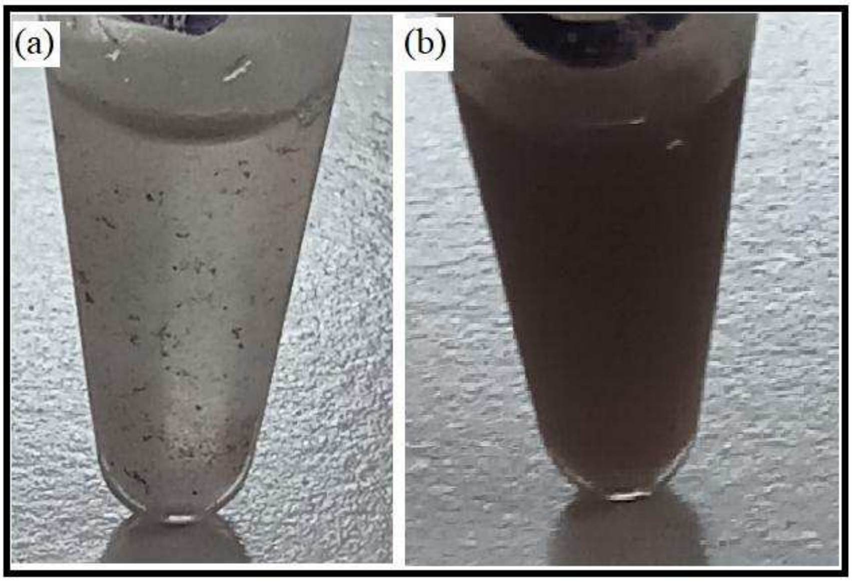


Figure 9: (a) Functionalized carbon nanospheres sticking to walls of container after sonication (b) Dispersed solution of bare carbon nano spheres.

Removal of solvent gave a black coloured solid that was dried in oven at 100℃ to remove water. After sonication of 60minutes, the functionalized CNS did not disperse like the initial CNS solution (**Figure- 9**).

RESULTS AND DISCUSSION

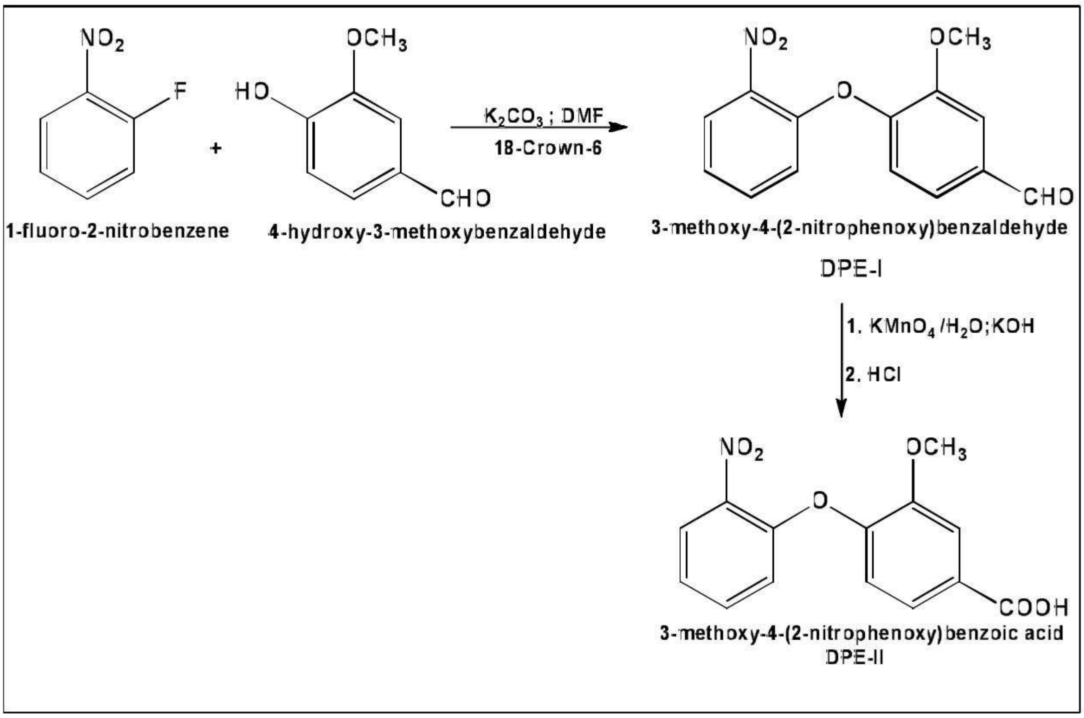
Material science, in last couple of decades, has evolved in a prolific way to provide many new materials. This include highly selective and accurate sensors, antibacterial surfaces[28,29] , food additive[30], therapeutic agent[31], cancer detection[32], and supercapacitors[33]. Despite tremendous progress in the material science, there is still a need to develop materials that can be used for the benefit of mankind. Carbon nanospheres (CNS) are the new materials that find wide applications in waste management[34], pollution reduction and purification of water[35]. However, their recovery after the process is still a challenge due their small size. Thus, there is need to work on the methodologies that facilitate their post process agglomeration and removal from the system.

Diphenyl ethers are organic molecules that are known for their antibacterial activity[36]. The use of this class of compounds for ion sensing has emerged in recent years[25, 26, 37,38]. However, the detection limit of these organic compounds for various ions is in micro molar range. Thus, there is need to improve their sensitivity for applications in real life. It was envisaged to tag organic molecules with the CNS and observe their behavior for various applications. The results below present tagging and characterization of diphenyl ether molecule with carbon nanospheres.

Synthesis of Diphenyl ether

Diphenyl ether **(DPE-II),** required for tagging with **CNS** was synthesized by a known procedure using nucleophilic aromatic substitution reaction[27].Briefly, 2-fluoro nitrobenzene and vanillin were reacted together in the presence of a mild base, potassium carbonate and 18-Crown-6 in aprotic solvent to get compound **DPE-I** (Scheme- II). The ^{1}H NMR spectra displayed a peak at δ 9.9 ppm for 1 proton and δ3.9 ppm for 3 protons corresponding to - CHO and -OCH_3 respectively. ^{13}C NMR spectra was also consistent with the structure of the compound displaying peaks at δ56.21 and 190.80 ppm corresponding to -CHO and - OCH_3 groups. **DPE-I** was then oxidized using $KMnO_4$ in aqueous media to get white solid compound **DPE-II** (Scheme II). The compound was characterized using ^{1}H and ^{13}C NMR spectra before proceeding to tag with CNS.

Table-I shows comparison of peaks for DPE-I and DPE-II. It can be seen that the peak at δ 9.9 ppm for -CHO group (in DPE-I) disappears in case of DPE-II due to conversion to corresponding acid in ^{1}H NMR.



Scheme II: Reaction scheme to synthesize Diphenyl ethers (DPE-I and DPE-II) for tagging with CNS.

Similarly, in case of ^{13}C NMR, spectra signal corresponding to -CHO at δ 190.8 ppm is shifted upfield to 169.8 due to -COOH. DPE-II obtained above was tagged to CNS by two methods as described below.

Tagging of DPEs to CNS

CNS synthesized by hydrothermal method were obtained as such after their characterization. Tagging of DPEs to CNS was done by two methodologies. First methodology involved ester bond formation by dehydration method with the hydroxyl groups present on the surface of CNS (**Figure 10**).

Here, DPE-II and aqueous CNS solution in ethanol were refluxed for five hours by dehydration method in the presence of catalytic amount of sulphuric acid (Scheme III). After cooling, the mixture containing black mass was centrifuged and washed with organic solvent (to remove any acid or organic compound) and water. The black coloured mass (DPE-III-A) was then proceeded for characterization.

^{1}H NMR		^{13}C NMR	
DPE-I (in ppm)	**DPE-II (in ppm)**	**DPE-I (in ppm)**	**DPE-II (in ppm)**
3.91	3.7	56.21	55.62
7.00	6.78	119.33	117.53
7.05	7.03	120.54	120.37
7.28	7.20	124.06	122.32
7.45	7.55	125.43	122.62
7.49	7.69	125.98	125.45
8.03	7.98	133.63	134.80
9.90	----	134.43	139.55
	.	140.91	143.15
		149.69	149.7
		149.85	150.60
		151.18	169.52
		190.80	169.63

Table-I: Comparison of δ values (in ppm) for DPE-I and DPE-II for ^{1}H ^{13}C NMR spectra

Two, by converting it to corresponding acid chloride and then making an ester by dehydrohalogenation. The acid chloride required for the purpose was prepared in-situ using phosphorous trichloride. Obtained acid chloride was dissolved in acetone and added to the aqueous solution of CNS containing acetone and potassium carbonate. Room temperature stirring for two hours followed by centrifugation gave black coloured solid mass. (Scheme III). This material (DPE-III-B) was washed with organic solvent (ethanol and acetone) to remove any unreacted DPE and potassium carbonate respectively. DPE-III-B was then characterized using various techniques.

Repeated attempts to disperse the solid mass by sonication up to 60 minutes were not successful. Conditions described above were optimized by using different ratios of DPE-II and CNS. As shown in **Table-II**, in both the methods, functionalization was successful when 0.172 mmol of DPE-II with 0.084 mol of CNS were used. The use of higher quantities did not give the desired results.

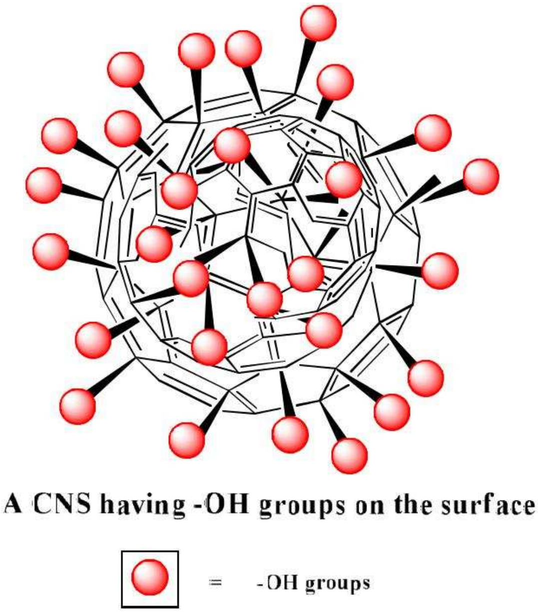
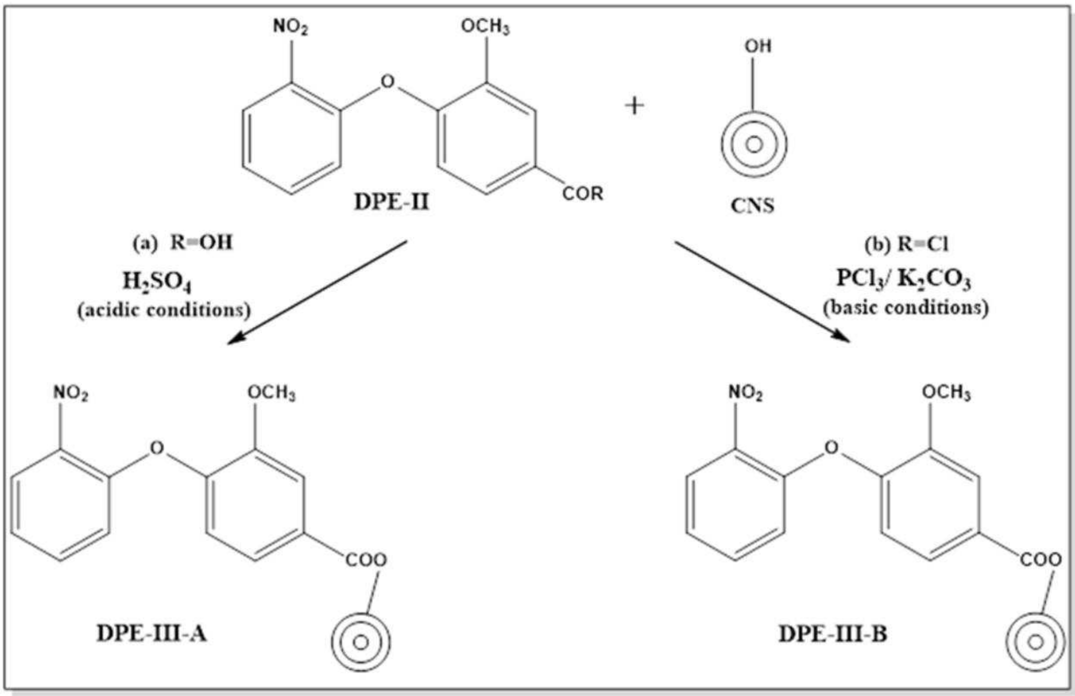


Figure 10: Cartoon diagram of carbon nanosphere (CNS) and various Hydroxyl groups present on its surface



Scheme III: Reaction to tag DPE II to CNS by (a) dehydration using acid conditions and (b) acid chloride using basic conditions. (K_2CO_3)

CNS amount taken		DPE-II amount taken		Method deployed (conditions)	Product formation
mmol	Volume (in ml)	mmol	Weight (in mg)		
0.08	3	0.68	200	Basic	No
0.08	3	0.68	200	Basic	No
0.08	3	0.17	50	Acidic	Yes
0.08	3	0.17	50	Basic	Yes
0.45	6	0.34	100	Acidic	No

Table-II: Comparison of ratios of CNS and DPE-II for product formation.

Characterization

Initial characterization of all the materials was done using Scanning Electron Microscope(SEM) technique. **Figure 11 (a)** shows SEM image of native CNS as obtained for functionalization. The approximate size of CNS is 470 nm as calculated. Morphological changes were observed in case of both DPE-III-A and DPE-III-B samples that were prepared by dehydration and acid chloride respectively as shown in **Figure 11 (b & c)**.

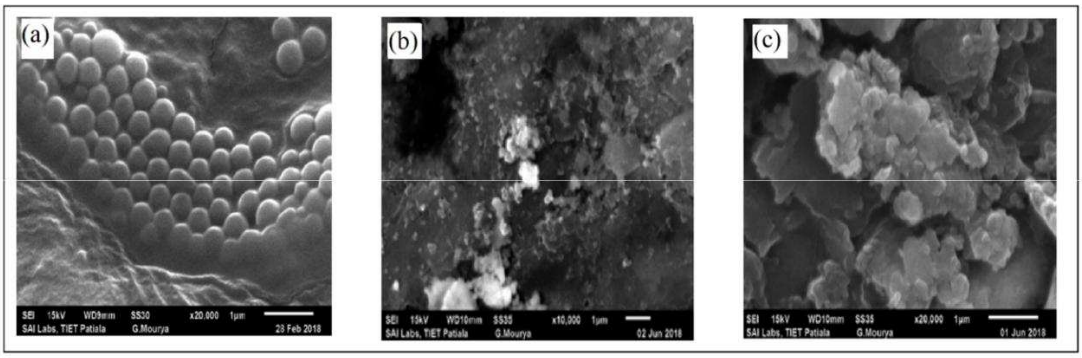


Figure 11: SEM images for (a) Sucrose CNS; (b) DPE-III-A and (c) DPE-III-B.

The optical studies using UV- Vis spectrophotometer showed absorption band at 248 and 296 nm for DPE-II and CNS respectively (**Figure 12-a & b**). After functionalization in both cases (DPE-III-A and DPE-III-B), a broad peak at approximately 264nm was observed (**Figure 12-c & d**). This blue shift w.r.t. CNS and red shift w.r.t. DPE-II can be attributed to shifting of electron density from CNS to organic moiety through the formed ester linkage.

This was also confirmed by the TAUC plot (**Figure 13 a-c**) where the band gap observed for bare CNS was 2.07 eV, which increased to 2.39 and 2.36 eV respectively after functionalization of CNS (**Table-III**)

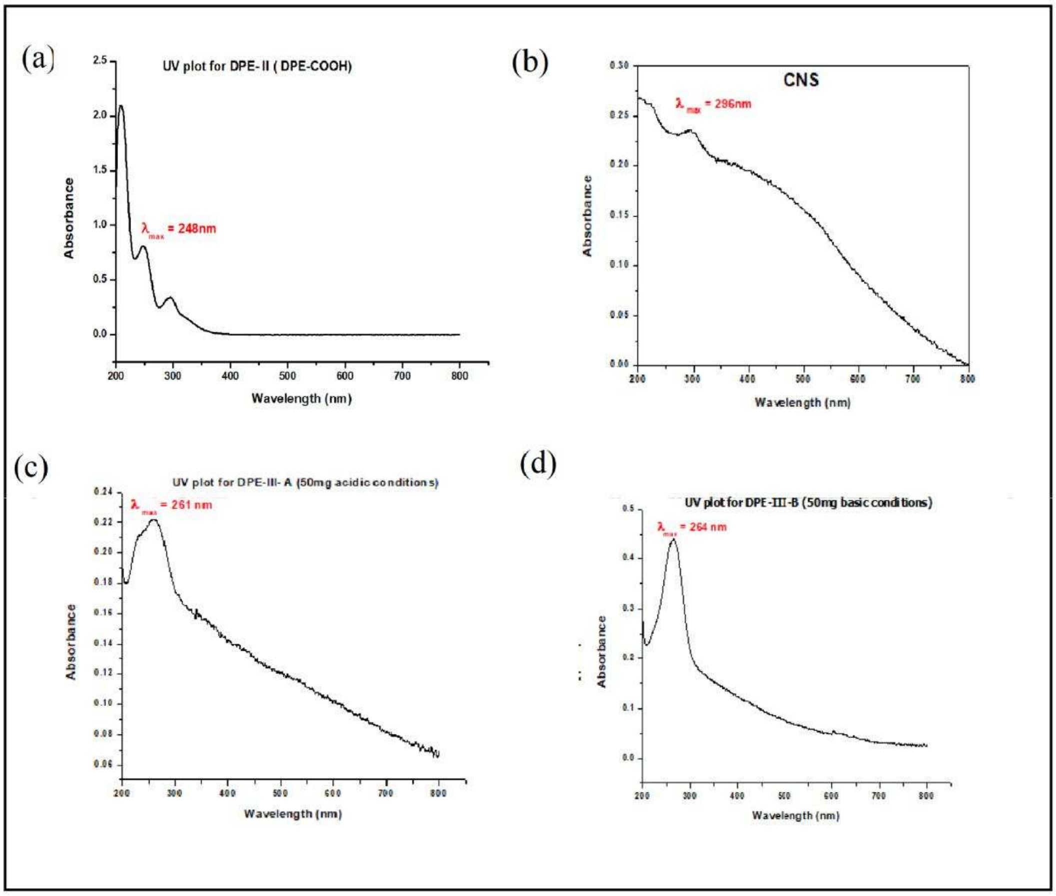


Figure- 12: shows the UV spectra of UV plot of (a) DPECNS;-II-II (b)DPECNS-II; (c) DPE-III-A and (d) DPE-III-B.

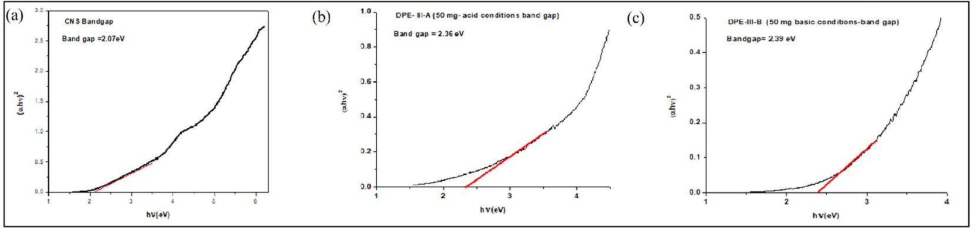


Figure- 13: TAUC plots of (a) CNS; (b) DPE-III-A and (c) DPE-III-B

Species	Band Gap (in eV)
CNS	2.07
(DPE-III) Using basic condition	2.39
(DPE-III) Using acidic conditions	2.36

Table- III: Summarized Band Gap of DPE-III (both using base and acid) and CNS.

Finally, the presence of the ester bond and other aromatic species was confirmed by ATR-IR technique. Carbonyl for the ester bond is expected to appear between 1730-1740 cm $^{-1}$. This characteristic peak that was missing in case of both the reactants (**Figure 14 a & b**), was observed in case of both DPE-III-A and DPE-III-B at 1736 and 1741cm $^{-1}$ (**Figure 14 c & d**) respectively along with other aromatic peaks.

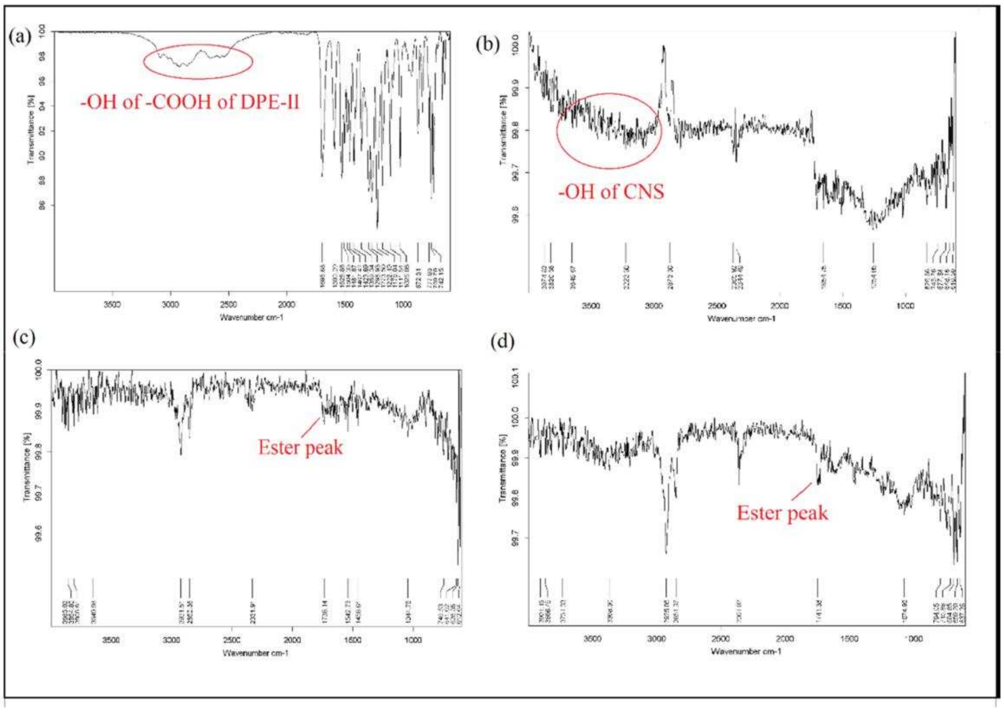


Figure- 14: shows ATR IR transmission spectra of (a) DPE-COOH; (b) CNS; (c) DPE-III-A and (d) DPE-III-B.

Thus, IR also confirmed the functionalization of DPE molecules on CNS. The work above has functionalized the CNS with DPE for potential applications in the waste management. The property that the CNS particles agglomerated after functionalization and could not be dispersed in the medium shows that functionalization on the CNS can be used to remove them from the system when centrifugation is not possible.

The work presented above shows surface functionalization of CNS with diphenyl ethers that results in former’s agglomeration. The functionalization was due to ester bond formation that was confirmed by IR and supported by morphological changes and their optical studies. The method may have applications in CNS post waste or adsorption treatment. The work described here can be extended to form antibacterial surfaces due to DPEs antibacterial properties.

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