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Ultrasonic Functionalization Of Graphene Oxide By Sulphanilic Acid And Used For The Selective Adsorption Of Methylene Blue Among The Cationic Dyes

S. D. Gedam[a], A.S.Rajurkar[a], C. S. Bhaskar[a], Sanjay R. Thakare[b]
[a]Department of Chemistry,Taywade College, Mahadula- Koradi, Nagpur 441111, India
[b]Department of Chemistry, Govt. Institute of Forensic Science, Nagpur 440001, India

ABSTRACT
Graphite oxide (GO) is oxygen containing derivative of graphite, in which it has various type of functional groups which are excellent chemical reactivity. Preparation of graphene oxide is done by Hummer's method, due to its highly chemical of reactivity of graphene oxide we can functionalized with different type of material. In this Research Work we report the use of sulphanilic acid as a surface modifying agent, sulphanilic acid incorporated on the graphene oxide surface. The –SO_3H functional groups of Sulphanilic acid were expected to provide surface compatibility form modified GO sheet. The product (S-Doped GO) which is confirmed by testing physiochemical analysis, surface analysis and elemental analysis bu various analytical techniques. S-doped GO selectively adsorbed methylene blue as a representative of cationic dye. This study shows the utility of this material for the separation and removal of methylene blue like dye which is environmentally very important.
Keywords: *Graphene oxide; Sulphanilic acid; S-Doped GO; Methylene Blue; Cationic dyes.*

1. INTRODUCTION

Water pollution is a critical issue confronting humanity, as wastewater presents significant threats to environmental integrity and human well-being. In this context, the treatment of colored wastewater has attracted significant attention. Dyes constitute an important category of synthetic organic substances. These substances have complex aromatic molecular frameworks, making them very stable and resilient to breakdown. Persistence, non-biodegradability, and toxicity are among the factors contributing to the detrimental impact of dyes on water and soil within the environment [1–2]. Different methods have been utilized for dye removal in water, including ion exchange, precipitation, osmosis, and so forth [3]. Nonetheless, owing to the method's straightforwardness and affordability, adsorption is extensively utilized.

The primary dye, methylene blue (MB), is used widely uses in the textile industry and also utilized in microbiology, diagnostics, and surgical procedures [3]. Even though it isn't very hazardous, it may produce detrimental impacts on human health, leading to eye injuries, elevated heart rate, diarrhoea, jaundice, cyanosis, quadriplegia, shock, and necrosis of tissue [3,4]. Therefore, there is an essential necessity to carry out studies aimed at removing this dye from water. A multitude of studies have thoroughly investigated the phenomenon of MB dye adsorption, utilizing a diverse range of adsorbents including natural and synthetic polymers, inorganic substances, soils, and additional elements [5].

Lately, focus has shifted towards the use of carbon-rich materials as adsorbents because of their availability, composition, chemical durability, and appropriateness for real-world use. Among these compounds, nanomaterials based on graphene, including graphene, graphene oxide (GO), and reduced graphene oxide (RGO), due to recent carbon modifications attracting significant interest in research investigation, because of their distinct architecture and extraordinary electronic and physicochemical properties [6-8]. In recent years, they have been utilized as advanced elements in numerous scientific and technological fields, such as nanomedicine and electronics, storage of energy, generation of power. The distinctive arrangement of graphene offers the chance for every carbon atom to be exposed at the surface and on both sides of the sheets is accessible to pollutant molecules. This substance has a distinct two-dimensional atomic lattice structure, consisting of a singular layer of tightly packed sp2 carbon atoms structured within a honeycomb structure comprises multiple kinds of functional groups on its surface such as carboxyl, carbonyl, alkoxy, epoxide, hydroxyl, and various oxygen-derived functional groups) [9-10]. These groups that contain oxygen are accountable for the potential. of the surface modification of GO, which offers numerous possibilities for employing graphene oxide utilized as a precursor in creating various nanocomposite materials. Vast Studies have shown the capability of graphene family nanomaterials as efficient nano-adsorbent materials. Consequently, these materials have been frequently described as

superior adsorbents. for the elimination of various contaminants, such as dyes, heavy metal ions, and radioactive materials.

The dominant approach for synthesizing graphene typically includes the oxidation of graphite, succeeded by exfoliation, leading to the creation of graphene oxide (GO). Every GO layer is regarded as a multifunctional network, comprising multiple oxygen functionalities and the carbon framework. Owing to its hydrophilic nature, GO can be effortlessly exfoliated and scattered in water or organic solutions. The hydrophilic nature of GO allows it to be uniformly distributed on the substrate in one layer only. The oxygen functional groups within the GO molecule create reactive sites for chemical modification. GO needs further functionalization to improve its thermal stability, dispersibility, and suitability with additional substrates [11-14]. To enhance its effectiveness for dye elimination, surface alteration of Graphene oxide (GO) is crucial. Pristine GO might be unhelpful, particularly concerning reusability, since many functional groups obstruct the dye's desorption from its surface. In this instance, the sulfonic acid group is crucial. Utilizing the solvation effect, this group can promote the separation of mobile protons, consequently improving ionic conductivity. In comparison to the carboxyl group, the sulfonated acid group exhibits superior effectiveness in proton dissociation and grants a greater ionic exchange capacity to the substrate because of its more robust acidity [15-18]. Certainly, the functionalization of GO with sulfonated acid groups can enhance ion exchange capability. and proton conductivity. The negatively charged functional groups of GO-SO3H are anticipated to enhance the adsorption capability through the electrostatic and dispersion interactions with the positively charged dye.

Consequently, we sulfonated GO through a reaction with sulphanilic acid under ultrasonication to create an improved adsorbent for the elimination MB. The effectiveness of the new adsorbent was assessed using experimental techniques, offering understanding into the adsorption process. Different methods, such as XRD, FTIR, SEM, Raman and XPS, were utilized to investigate the microstructure-property correlation of the product. The study validates the effective alteration of sulfonated graphene oxide for the adsorption of the cationic dye.

2. EXPERIMENTAL SECTION.

Graphite powder was obtained from Sigma Aldrich, Sulphuric acid, hydrogen peroxide, potassium permanganate, solid 2-aminopyridine other reagents were of analytical grade (Merck) and used as received without any purification.

2.1 Preparation of Graphene Oxide

Graphene oxide (GO) was synthesized according to modified Hummers and Offerman method from purified natural graphite powder. we prepared Graphene oxide nanosheets by well-known modified Hummers' method. The method was described briefly as follows. Graphite powder (1.0 gm) and sodium nitrate (0.5 gm) were added in a 23 mL concentrated sulphuric acid in a round bottom flask (500 mL), placed in an ice-bath. The temperature of ice bath should not exceed 15^{O}C. 3 gm potassium permanganate was added slowly by continuous stirring for 2 hours. This solution was transferred to a beaker and kept it in water bath having temperature 35^{O}C and stirred for next 30 minutes. After that, distilled water (46 mL) was slowly added into the above solution. The temperature of the solution was carefully monitored at 9^{O}C during the addition of distilled water. After the complete addition of distilled water, the temperature of the solution was maintained at 98^{O}C for 30 minutes. To terminate the reaction, 140 mL distilled water and 10 mL of hydrogen peroxide was added in the above solution. The color of the reaction mixture was looking like dark yellow. The reaction mixture was filtered and washed with 5% HCl solution, followed by distilled water in instalment for several times. The filtered product was kept for 24 hours at room temperature to get powdered graphene oxide. Complete drying is further achieved by keeping the product at 60^{O}C in oven for 1–2 hours.

2.2 Synthesis of S-Doped Graphene Oxide

Herein, we reveal utilization of sulphanilic acid as a surface modifying agent, sulphanilic acid incorporated on graphene surface via p–p interaction. Functional groups like –SO3H of Sulphanilic acid have been anticipated to give surface compatibility form modified SA-GO sheet. GO has been functionalized with sulphanilic acid, where the product was obtained by the same simple dispersion chemical method. 0.5g of GO has been dispersed in 50ml water by using ultra sonication for half an hour in a beaker, later 1.73g and 10m mole of sulphanilic acid has been mixed with 10 ml slightly cold water then GO solution and sulphanilic acid solution were mixed as well as dispersed by ultra sonication for 30 minutes. Suspension has been stored under magnetic stir for 30 minutes. After that suspension has been heated under water

bath for 1 hr. at 80°C. Afterward suspension was rinsed numerous times with distilled ethanol and water. At last stage filtration done by simple glass funnel. Crude product gained has been dried up at 70°C for 6 hours to gather black powder product and here after labelled as S-Doped GO. This is represented in figure 1.

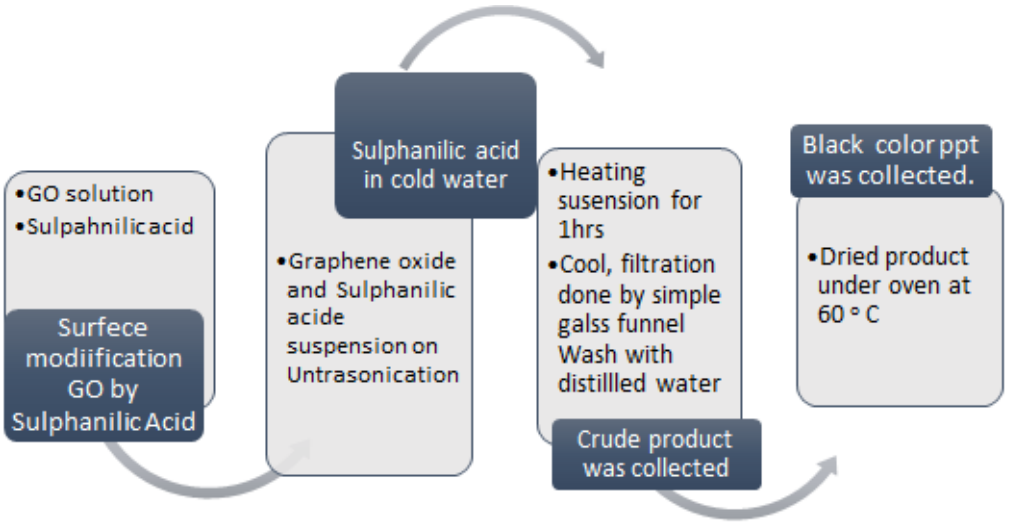
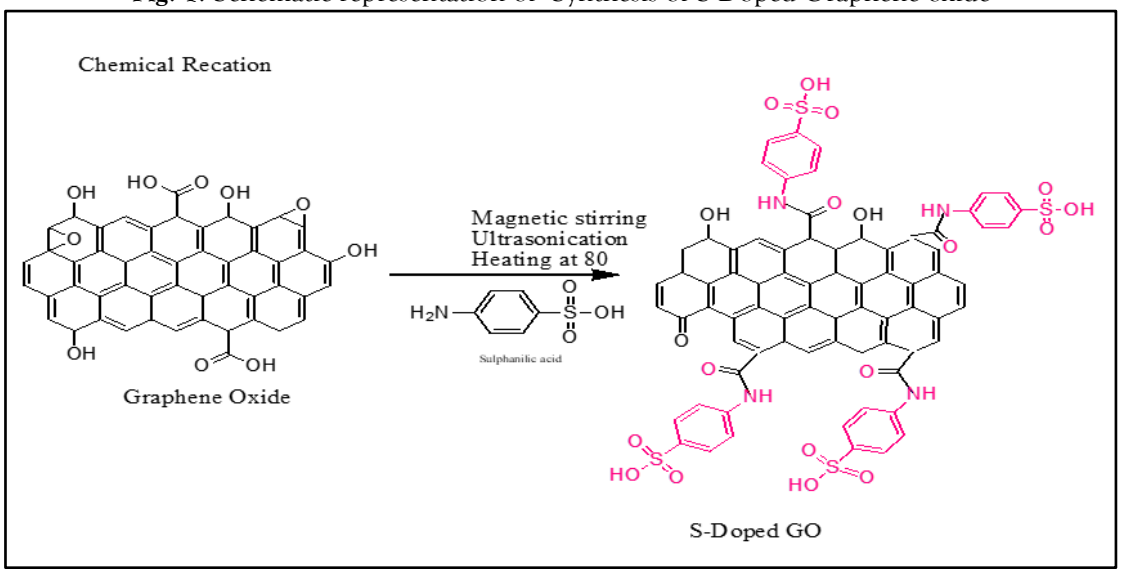


Fig. 1. Schematic representation of Synthesis of S-Doped Graphene oxide



Scheme-I: Schematic representation of the Synthesis of S-Doped graphene oxide (S-Doped GO)

3. RESULTS AND DISCUSSION

3.1 Characterization of materials.

The prepared GO and its functionalization with sulphanilic acid confirmed using FT-IR and results are represented in Figure 2. FTIR Spectrum of For Pure Graphene oxide shows a broad peak appeared at in the range of 3339 and 3304 cm^{-1} in the high frequency area attributed to the stretching mode of O-H bond, reveals the presence of hydroxyl group in Graphene oxide. The Sharp peak at 1620 cm^{-1} for C=C bond in Benzene ring. The peak at 1041 cm^{-1} corresponds to vibrational mode of the of the ν_s(C–O) bond. In middle of the FTIR Spectrum of GO shows sharp peak at 2113 cm^{-1} of O-C=C bond. From above fig. 2b FTIR Spectrum of S-doped GO exhibit, peaks present at 1286 & 1340cm^{-1} are critically attenuated in S-doped GO. The main peak seen at 1116cm^{-1} (S-O) bond and 1047 cm^{-1} (S-phenyl) confirm the existence of -SO_3 sulphanilic acid group. Other peaks at 947 cm^{-1} for C-H in plane deformation and at 815cm^{-1} out of plane C-H wagging are distinguishing vibrational peaks of a p-di substituted phenyl groups.

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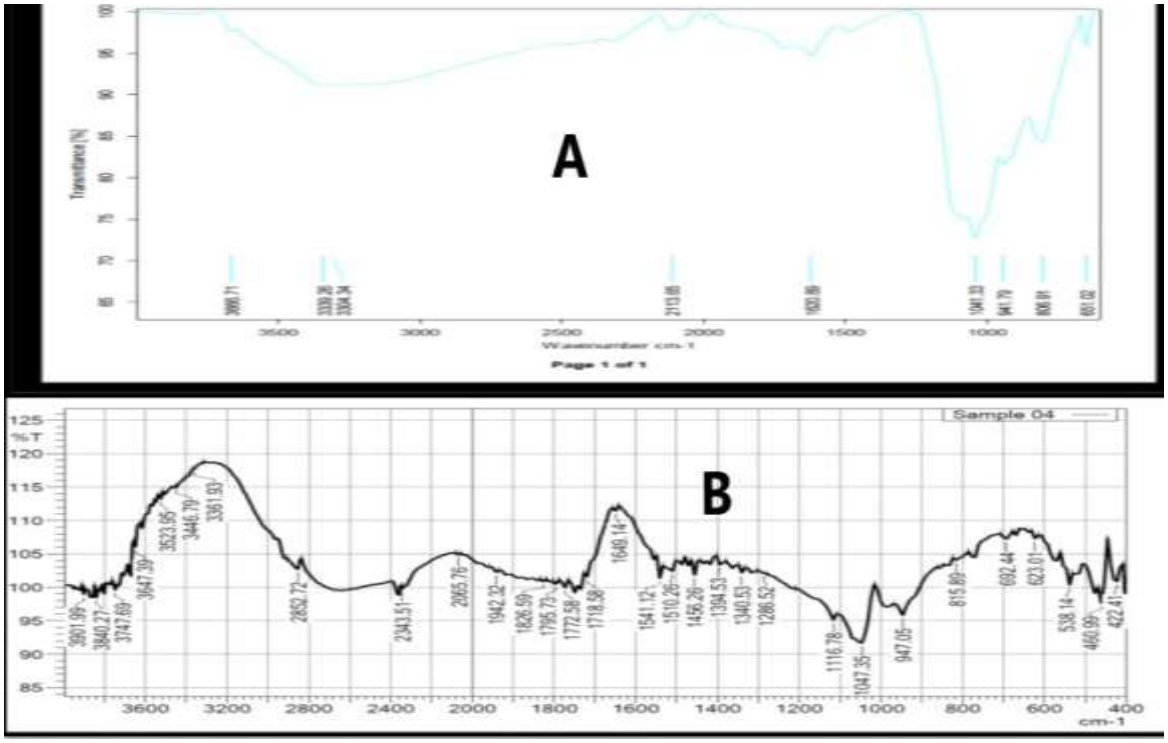

Figure 2: FTIR Spectra of graphene oxide and S-doped GO composite (A) GO and (B) S-doped GO

The functionalisation of GO by sulphanilic acid take place was confirmed using the FTIR analysis and further it confirms using XRD analysis. Because functionalisation changes the structural changes in the molecule and hence to observe the structural changes, XRD analysis of GO and S-doped GO was carried out. The XRD pattern shown in Fig. 3 a represents the sharp diffraction peak at 10 °indicates (001) plane of the pristine GO in agreement with literature [19]. The diffraction peaks clearly seen 2θ value of 10.20° in the XRD pattern of Prepared Graphene oxide arises because of the diffraction from the different oxygen containing functional groups on the surface of the graphene oxide sheet. From figure 3B XRD pattern of S-doped GO composite, shows two diffraction peaks at 2θ=10° and another at 2θ=28, 2θ=30° which indicate that successfully non-covalent attachment of sulphanilic acid through S- GO linkage and increases intensity of two peaks supported the S – doped GO was successfully prepared.

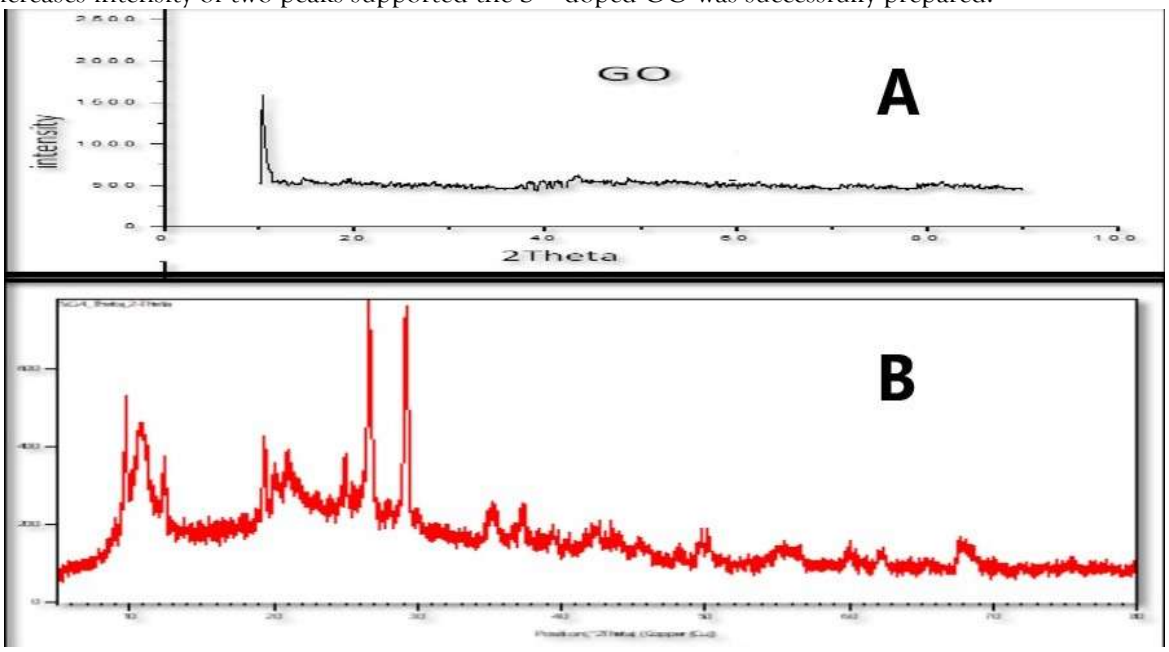

Figure. 3. XRD Pattern of GO and S-doped GO composite

Morphological analysis of resultant material of GO with functionalization with sulphanilic acid was investigated. SEM morphology of prepared GO and their composites have been investigated by SEM at several magnifications. Demonstrates in Fig.4a, 4b, 4c,4d, 4e and 4f) of GO.

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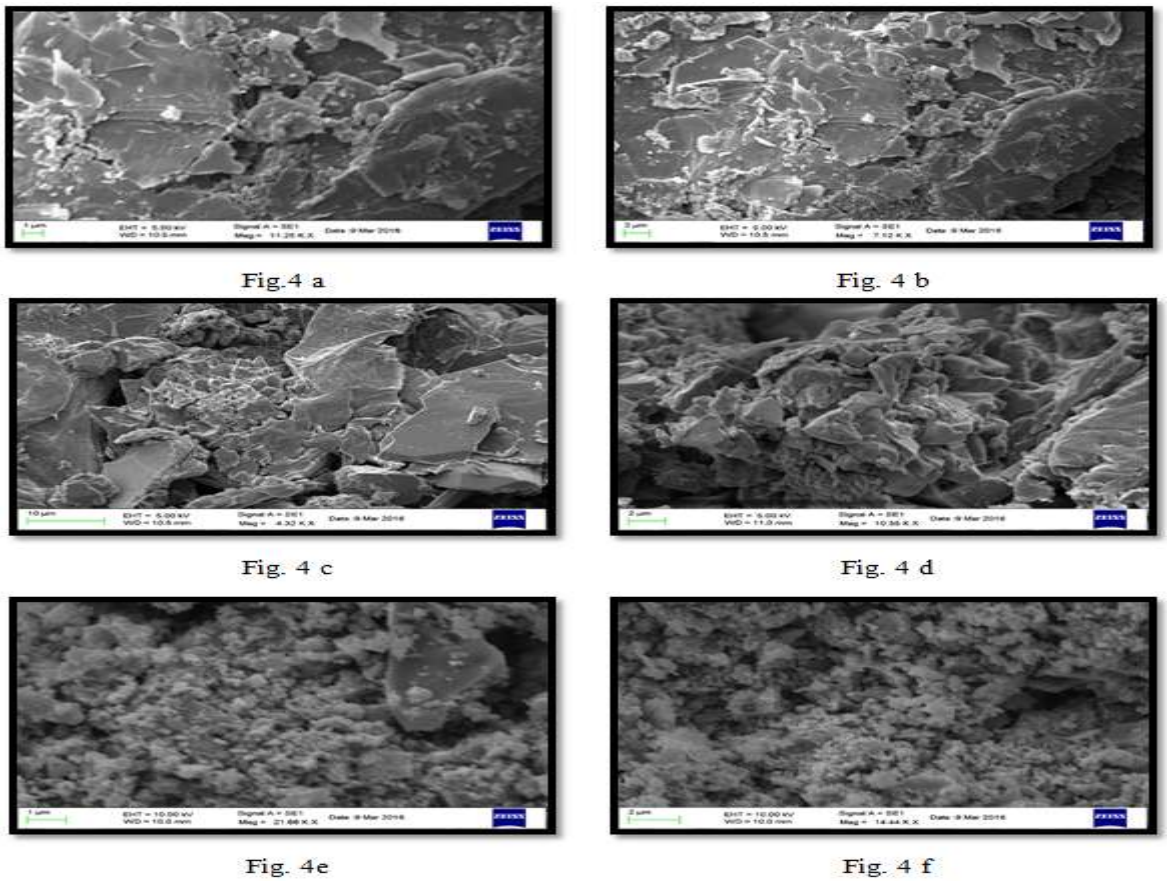

Fig.4 a Fig. 4 b

Fig. 4 c Fig. 4 d

Fig. 4e Fig. 4 f

Figure 4 (a-f): SEM Analysis of GO and S-doped GO composite

SEM analysis of graphene oxide in previous studies indicates crumpled morphology of densely packed sheets of GO. GO formed exposed thin flex and layered nanosheets that might facilitate the passage of electron beams. Additionally, GO nanosheets exhibited crumple and ripple structures, outlined in [20]. These characteristics may have appeared as a result of deformation of GO layers that transpired during exfoliation and restacking procedures.

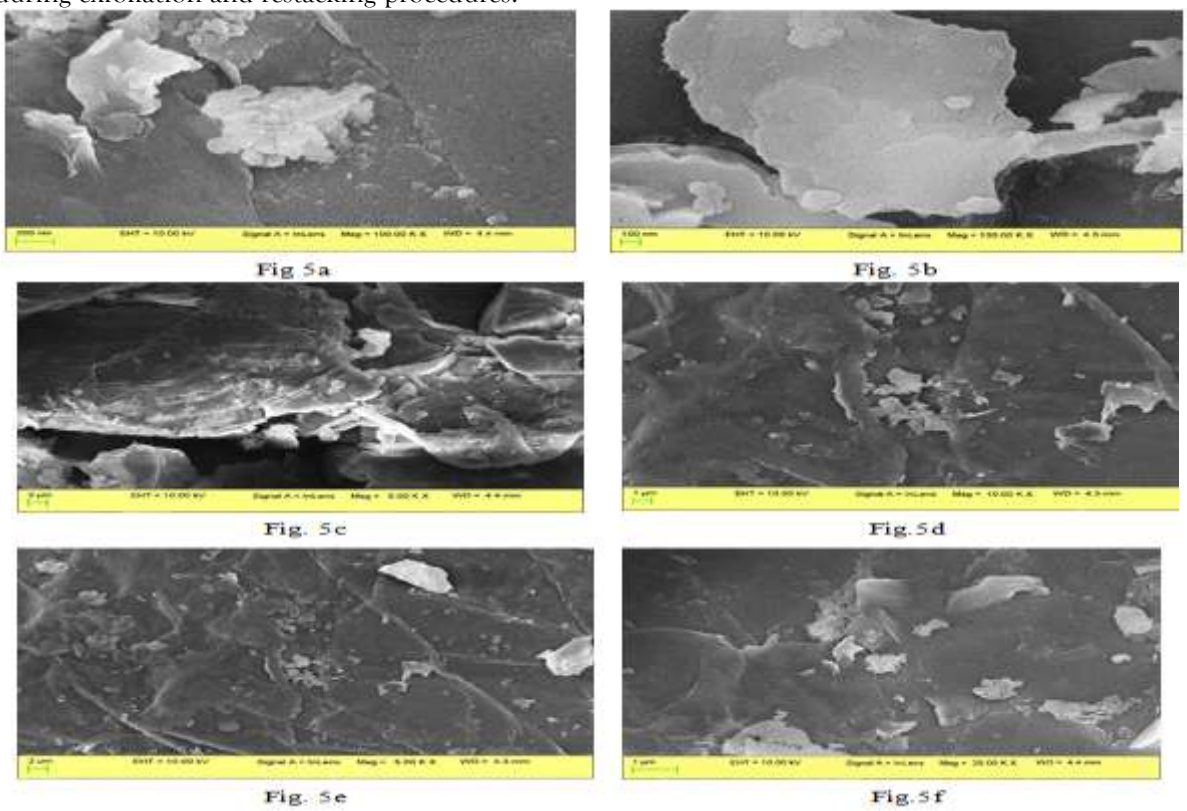

Fig 5a Fig. 5b

Fig. 5c Fig. 5d

Fig. 5e Fig. 5f

Figure 5 (a-f): SEM Images of S-doped GO Composite

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In S-doped GO composite SEM Analysis shows "figure 5a, 5b, 5c, 5d, 5e and 5f" shows that, few light-colored layers indicate re-stacked layers, while others are dark grey. Crumpled, rippling layers have sharp edges. C and O elemental composition of graphene oxide (GO) changes when N and S from sulphanilic acid are loaded. Thus, the compounds were successfully synthesized in this investigation.

Table I: Elemental analysis of S-doped GO.

Elements	Weight %	Atomic %
C K	37.82	44.88
N K	4.65	4.74
O K	55.61	49.53
S K	1.92	0.85
Totals	100.00	

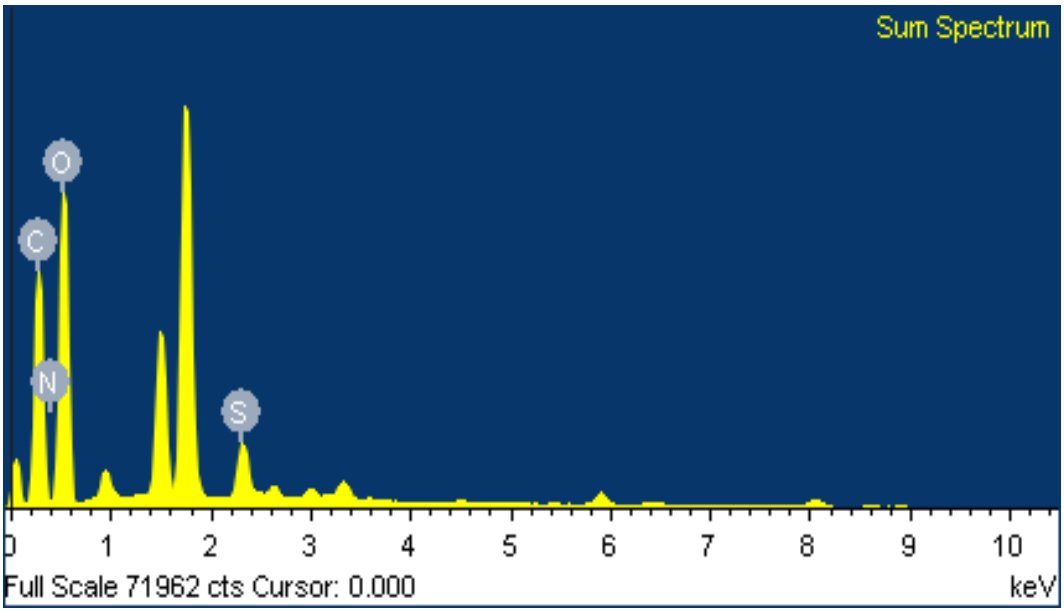


Figure 6: Electron Analysis of S-doped GO

From above elemental analysis of S-doped GO composite table 1, figure 6 (electron image of S-doped GO) after functionalization of GO, concentration of oxygen and carbon become 55.61 atomic, 49.53% weight and 37.82 atomic, 44.88% weight. Apart from this table I clearly shows that new heteroatom was introduced in the composite material from sulphanilic acid (sulphur and nitrogen) in S-doped GO composite. Concentration of Sulphur and Nitrogen were found little amount, 1.92 weight and 0.85% atomic, and 4.65 weight and 4.74% atomic, respectively.

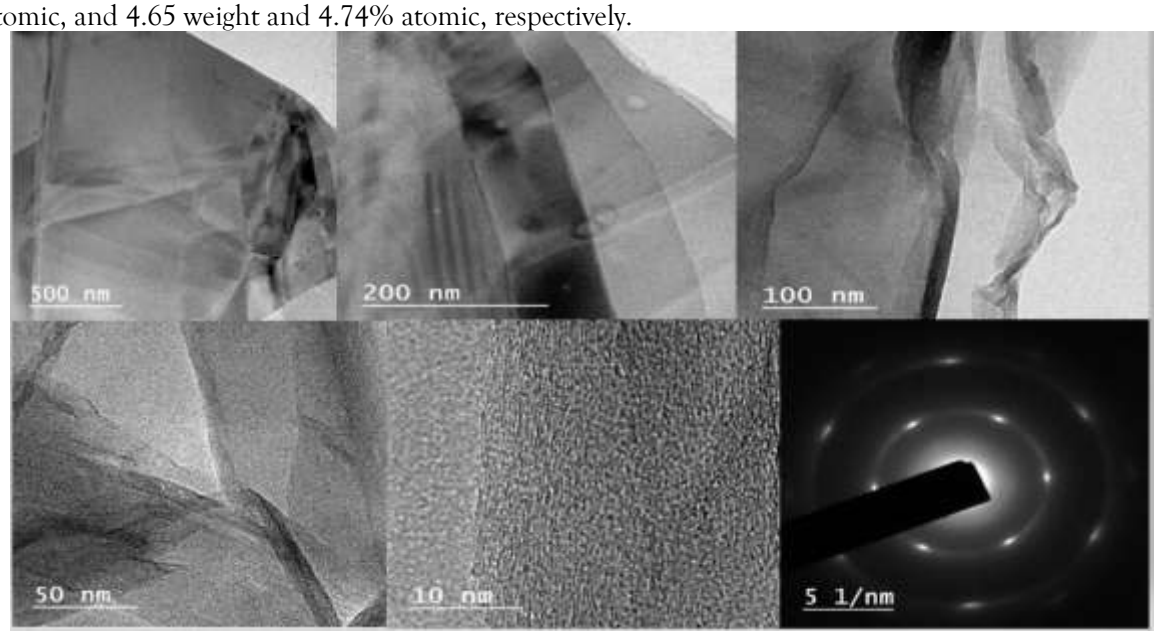


Figure 7: TEM images of S-doped GO Composite

TEM images of S-doped GO composite figure 7 demonstrate excellently. TEM revealed thin two-

dimensional sheets exhibiting numerous wrinkles, indicating considerable exfoliation of GO sheets during surface functionalization. SAED indicated S-doped GO. SAED pattern image of S-doped GO displays a ring, indicating polycrystalline nature due to GO grain overlap.

AFM is a versatile instrument that enables visualization of a specimen's topography, measuring its roughness, and identifying various phases of a composite. [21, 22]. Resulting GO and decreased GO nanosheets had typical thicknesses of around 1.0 and 0.8nm, respectively. AFM photograph of prepared GO represented in figure 8 and it is observed that the carbon sheets are overlapped on one another form a clumsy structure results in to the formation of rougher surface.

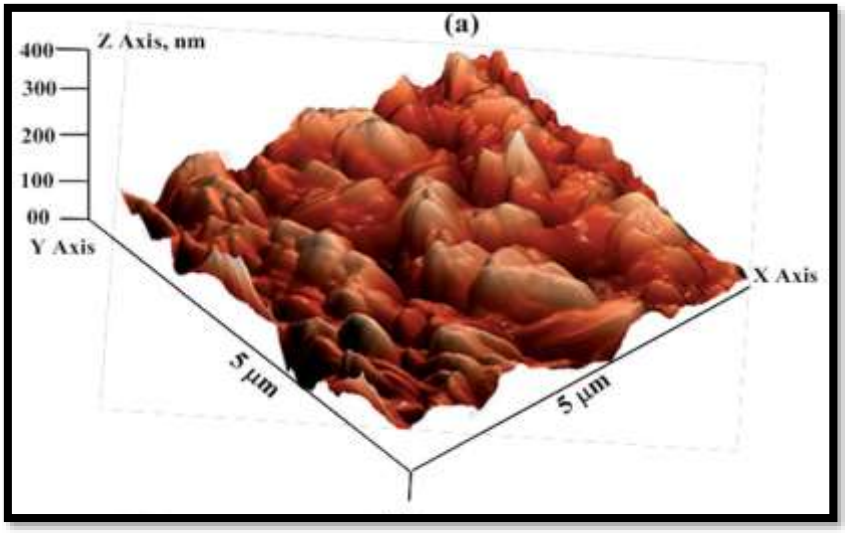


Figure 8: AFM Micrograph of GO

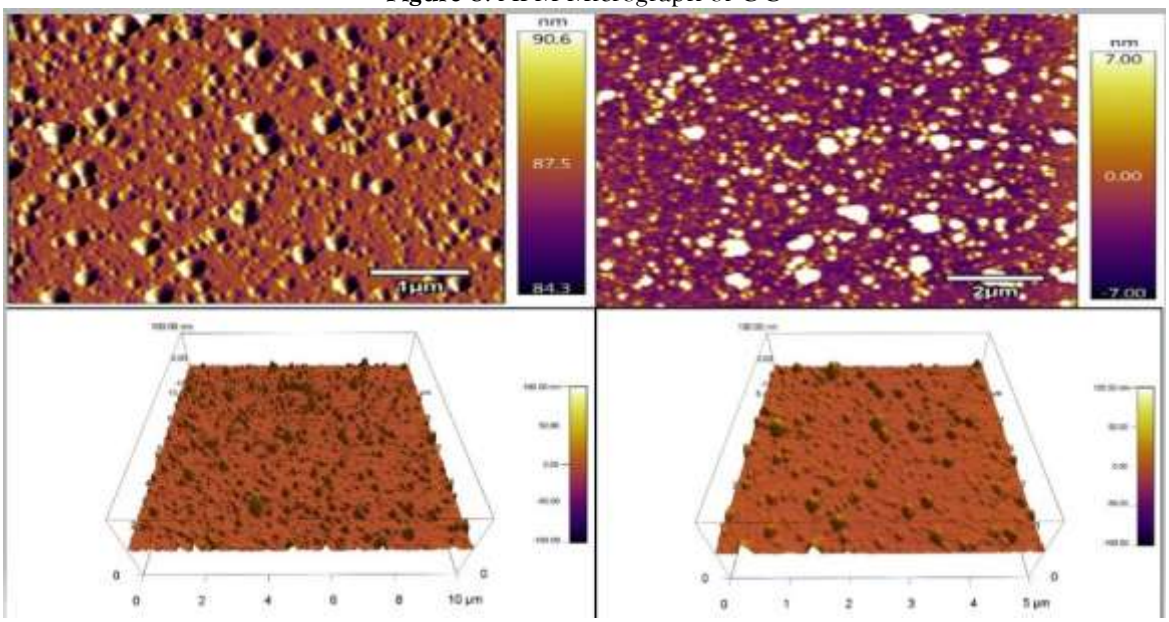


Figure 9: AFM Micrographs of S-doped GO composite

From above figure 9 of S-doped GO shows different AFM Micrograph shows sulphanilic acid successfully interacted on large surface area of GO which makes some spike shape structure on surface of GO indicate exfoliated GO sheets randomly distributed. From the comparison of Go and S-doped GO, S-doped GO has less height than GO indicates there is separation of carbon layers after the functionalization of GO with sulphanilic acid.

To analyzed the introduction of S and N in to the structure of GO after the functionalization, we carried out XPS analysis and results are represented in table II and figure 10. Data of table II and figure shows that after the functionalization of GO with sulphanilic acid was happed successfully.

Table II: XPS analysis of S-doped GO.

Element	Position	FWHM	Area / (T*MFP)	%At Conc.	% Mass Conc.	Mass
C 1s	283.87	3.7383	437.593	69.48	61.52	12.011

N 1s	400.51	2.5975	47.775	4.21	4.35	14.0067
O 1s	531.77	2.5254	436.955	23.68	27.93	15.9994
S 2p	167	0.8338	27.745	2.62	6.2	32.0648

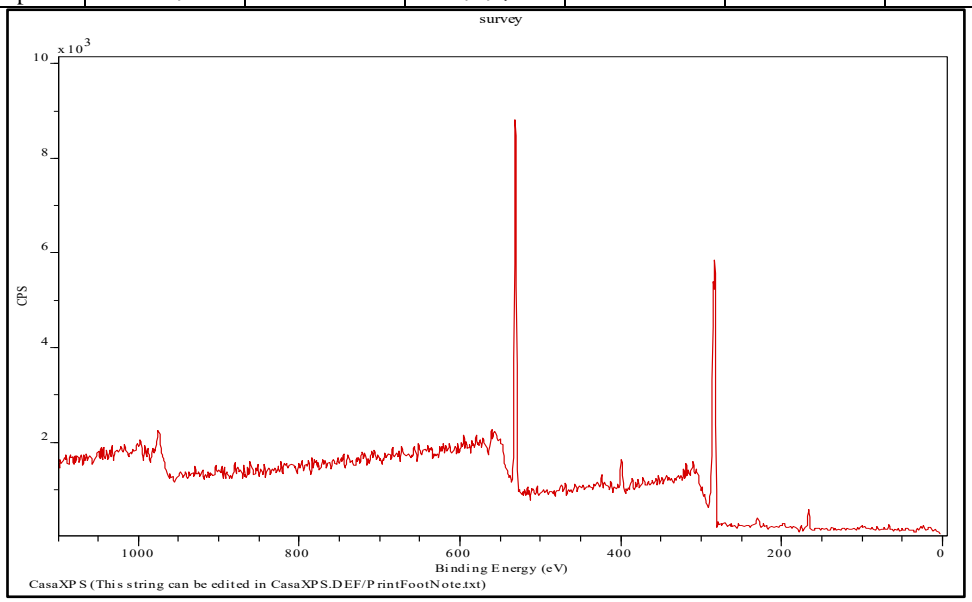


Figure 10: XPS Spectra of S-Doped GO

All above spectral data evident that the functionalization of GO by various materials were successfully carried out.

3.2 Application of S-doped GO for the MB separation

The characterization of S-doped GO was carried out and confirm the incorporation of sulphanilic acid in the network of graphene oxide which is schematically represented in the scheme I. this representation shows that the graphene oxide surface was richer to the negative charge as the sulphanilic acid is incorporated in the network. To find out the utility of the prepared S-doped GO, we carried out adsorption of dyes. For the comparison we choose two cationic and one anionic dye. Methylene blue and Rhodamine Blue are selected as a member of cationic dye as a representative and Methyl orange as an anionic dye as a representative. The adsorption of dye at fix S-doped GO and at same pH of solution was carried out for these three dyes molecules and results are represented in the figure 11 and table III

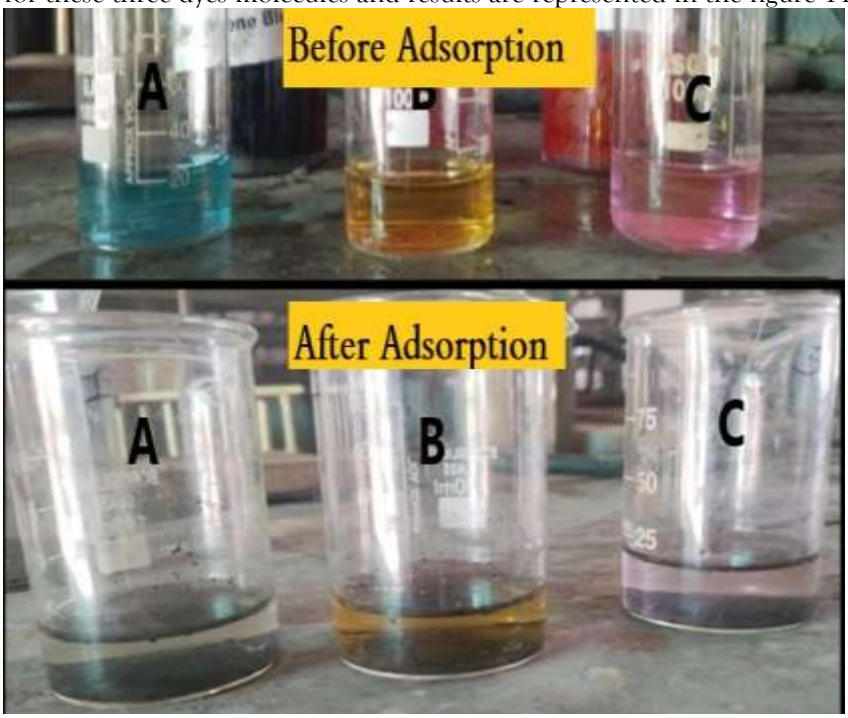


Figure 11: Adsorption of Dyes solution using S-doped GO Composite (A) Methylene Blue; (B) Methyl Orange; (C) Rhodamine B

Table III: Adsorption of Dyes solution with S-Doped GO Composite (A) Methylene Blue; (B) Methyl

Orange; (C) Rhodamine B.

Beaker Number	Name of Dyes	Amount of S-Doped GO	Total concentration	Colour	Time taken in min	observation
1	Methylene Blue	5 mg	10 ppm	Blue	3 min	Almost Adsorbed blue colour becomes colourless
2	Methyl Orange	5 mg	10 ppm	Orange	3min 50 sec	No change
3	Rhodamine	5 mg	10 ppm	Pink	10 min	No Adsorption slight pink colour change

From table III and figure 11, It is observed that our composite selectively adsorbs methylene blue dye completely while small amount of Rhodamine blue dye adsorption was also observed. While methyl orange was not adsorbed by at all. This result concluded that the prepared S-doped GO adsorb cationic dyes while no adsorption of anionic dyes. Among the cationic dyes, methylene blue is selectively adsorbed by the S-doped GO over the rhodamine blue. This is not only observed in the individual solution of dyes but similar results are observed when we used the mixture of dye solution. It is reported that, in acidic conditions, Methylene Blue is protonated and carries a positive charge, but at neutral or alkaline pH, it exists as the less charged or uncharged form. Similarly at acidic pH, rhodamine B structure can shift to a more unstable quinoid form or undergo N-de-ethylation. While in case of methyl orange, benzenoid forms in basic conditions is observed and red, quinonoid forms in present in acidic conditions. So, this concluded that the S-doped GO has a more negative charge and hence positively charged methylene blue adsorb immediately while Rhodamine B is a cationic dye but quinoid structure does not fascinate the adsorption. Similar reason may be for methyl orange dye.

For further investigation, methylene blue adsorption by S-doped GO was studied at different pH and results are represented in figure 12.

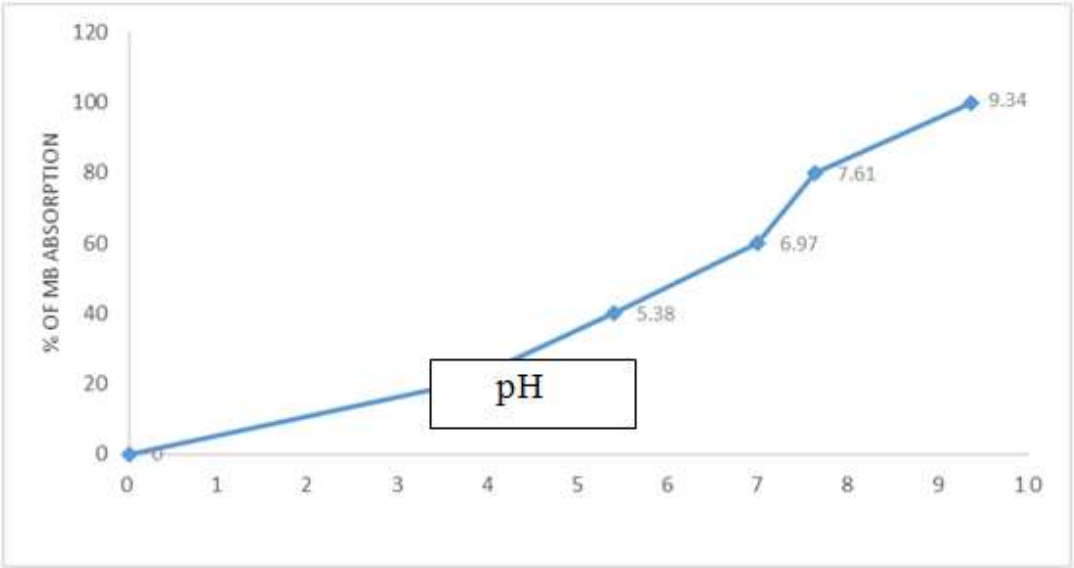


Figure 12: Adsorption capacity MB by S-Doped GO at different pH

Figure 12 shows that the adsorption capacity increases with increases from pH 3.70, 5.38, 6.97, 7.61 & 9.34 respectively. This indicate that there is a competitive adsorption site on the materials for the dye molecule as well as proton.

As the pH increases the protonation of S-doped GO increases due to abstraction of proton from material leads to the formation of more negative charge on the surface of material which fascinate the adsorption of methylene blue having positive charged.

The pH effect could be divided into two regions and explained by electrostatic interactions: (i) In first region (3.70≤ pH ≤ 6.97), cationic MB dye molecules are adsorbed with typical acidic cations, (ii) second region (7.61 ≤ pH ≤ 9.38) is neutral as cations decrease with increasing pH, allowing only cationic dye molecules to interact with S-Doped GO composite. Near pH 10 or higher, excess oxygens (OH− ions)

interact with cationic dye molecules, maintaining them suspended in the solution.
The adsorption of methylene dye with different amount of S-doped GO was also studied and it is observed that as the amount of S-doped GO increases, the methylene blue dye adsorption was also increased in a stated amount of S-doped GO up to 15 mg. The result is represented in figure 13.

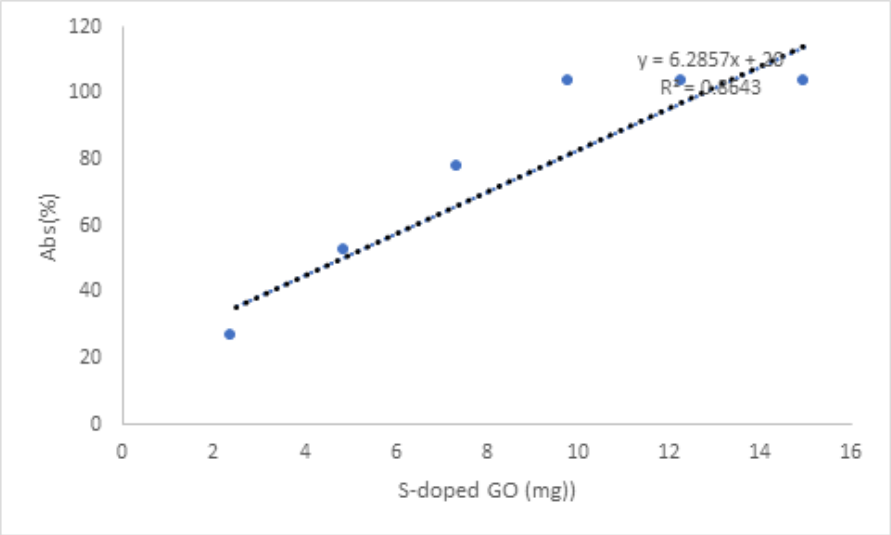
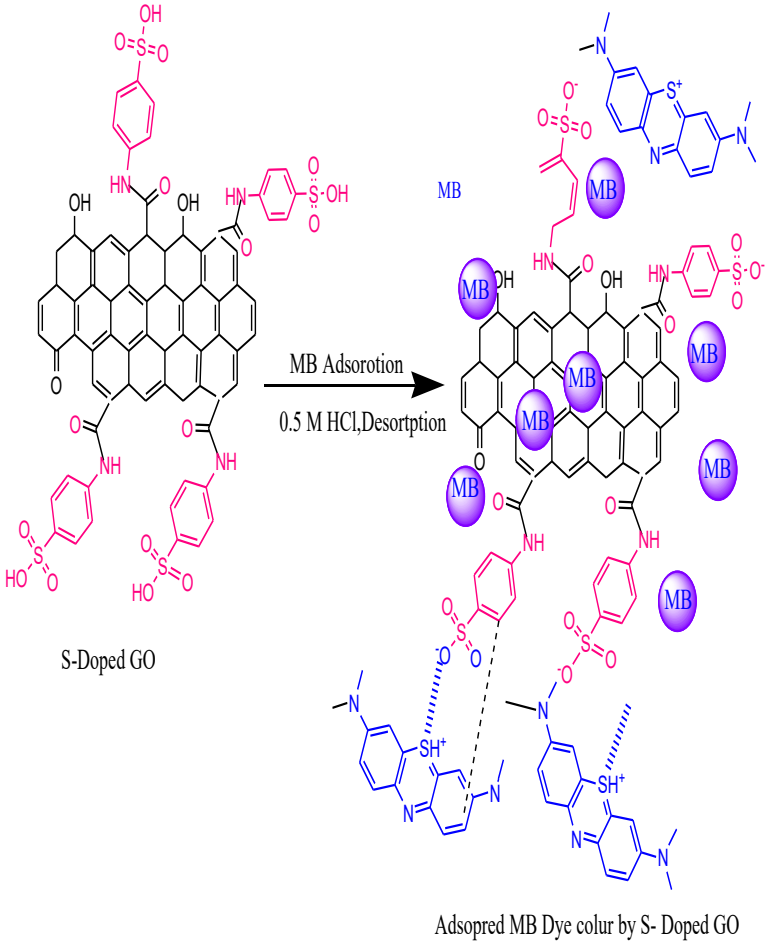


Figure 13: Adsorption of MB with different dosage of S-doped GO. Conc of MB-10 ppm; Time- 3 min, pH of solution-5

The results represented in figure 13 support our hypothesis. On the basis of experimental evidences and earlier report, the methylene blue dye adsorption on S-doped GO is represented in Schemce II



Scheme 2: Schematic representation of the adsorption of MB dye by S-Doped GO

CONCLUSION

The present study involves in the functionalization of graphene oxide by Sulphanilic acid namely synthesized as S-Doped GO. and their physiochemical study done by using - FT-IR, XRD, AFM, Raman ,SEM-EDX , TEM Raman spectra and XPS methods. Morphological and Surface Analysis done by SEM-EDX and TEM of GO and S-Doped GO. Shows very different morphology of Before and after functionalization of Graphene oxide with Sulphur functional group also signified new developed small grey like particle seen on structure in GO sheet which was seen in TEM Analysis. EDX provide new content of element which was absent in pure graphene oxide but after functionalization, it was found that Sulphur and nitrogen elements in S-Doped GO which can evident that successfully S-doped on Graphene Oxide sheet. AFM determines the dimensions and roughness of GO and S-Doped GO which was almost shows different roughness of GO and S-Doped GO. From XPS data and Raman spectra also supported the successfully functionalization of Graphene oxide by Sulphanilic acid. From Experimental data Prepared S-Doped composite show selective adsorption capacity toward MB Dye.

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