Photocatalytic Properties of GO/CdS/(Cd_{0.8}Zn_{0.2})S/TiO₂ Binary and Ternary Nanocomposites

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Abstract: GO-CdS, GO-(Cd_{0.8} Zn_{0.2})S, GO-TiO₂, GO-CdS-(Cd_{0.8} Zn_{0.2})S, GO-CdS-TiO₂ binary and ternary nanocomposites were prepared by chemical bath precipitation method at 80°C for 2 hours and their photocatalytic properties were investigated. The measurement of photocatalytic degradation of Rhodamine B dye was carried out under visible light for single materials as well as binary and ternary composites. The photocatalytic efficiency of the synthesized nanocomposites was calculated and was found to be higher for the ternary nanocomposites in comparison to binary and single materials. The nanocomposites were further characterized by XRD, SEM and EDX studies. XRD studies show planes of CdS and ZnS in binary nanocomposites while in the ternary composites, planes corresponding to CdS, ZnS and TiO₂ are observed. SEM images show a non-uniform distribution of spherical particles forming clusters on the surface of GO sheets. EDX spectra confirm the presence of C, O, S, Cd, Zn and Ti with excess Carbon. The studies suggest that the synthesized ternary nanocomposites are quite efficient materials as adsorbents for photocatalytic degradation of dyes from waste water.

Index Terms: GO, photocatalysis, binary and ternary nanocomposites, waste water treatment.

I. INTRODUCTION

Nanotechnology for water and waste water treatment is gaining momentum globally. The unique properties of nanomaterials and their convergence with current treatment technologies present great opportunities to revolutionise water and waste water treatment. An important technology that shows most promise in full scale application in the near future is photocatalysis.

The II–VI semiconductor, particularly CdS, is a good candidate for photocatalysis of organic dyes from water. Although the photo generated electrons and holes play a vital role in photo catalytic disinfection, their recombination result in low efficiency of photo catalysis. Hybrid of CdS with some support materials like Graphene/Graphene Oxide (GO) can delay the recombination process as well as adsorb the pollutants [1,2,3,4].

There have been reports that nanocomposites composed of CdS/ZnS and Graphene/Graphene Oxide showed significantly improved photocatalytic properties. Studies have also shown that incorporation of Graphene/Graphene Oxide with appropriate ratio of CdS to ZnS causes improved charge separation and enhanced visible light absorption and also exhibited a red-shift in band-edge compared to Graphene–CdS composites [5]. Several methods like hydrothermal, solvothermal, CBD, microwave method have been employed to synthesis these nanocomposites. However, much work has not done in achieving a cost-effective method in their synthesis.

Some researchers have also reported a ternary photocatalyst system such as $rGO/TiO_2/ZnO[6]$, $rGO/CoFe_2O_4/TiO_2[7]$, $rGO/Ag/TiO_2/Fe_2O_3[8]$, rGO/CdS/ZnO[9], and $rGO/W/BiVO_4[10]$. The competency of the ternary system was investigated and compared using a binary system (coupled with GO) and single photocatalyst material. The degradation of the ternary system was superior as confirmed by [6,8,9]. There was an improved surface area for the ternary system nanocomposite, which resulted in an increased adsorptivity, as reported by [6,7].

There have been no reports so far of synthesis of $GO/CdS/(Cd_{0.8} Zn_{0.2})S/TiO_2$ binary and ternary nanocomposites by the simple low cost and low temperature technique of chemical bath precipitation. The present paper reports the synthesis of $GO/CdS/(Cd_{0.8} Zn_{0.2})S/TiO_2$ binary and ternary nanocomposites by chemical bath precipitation method at 80°C for 2 hours and their photocatalytic properties were investigated. A comparative study of the photocatalytic behaviour of single material as well as the binary and ternary nanocomposites was done and the photocatalytic efficiency was calculated. The materials were further characterized by XRD, SEM and EDX studies.

II. EXPERIMENTAL TECHNIQUES

2.1 Synthesis of Graphene Oxide by Modified Hummer's Method:

Graphene oxide was produced from pure graphite using modified Hummer's method [11]. 108ml of conc Sulphuric acid (H_2SO_4) and 12ml of Phosphoric acid (H_3PO_4) in the volume ratio of 9:1 were mixed and stirred for several minutes. While stirred, 0.9g of graphite powder was added followed by slow addition of 5.28g of Potassium Permanganate (KMnO₄). The mixture was stirred for 6 hours until the solution turned dark green.2.7ml of hydrogen peroxide (H_2O_2) was added drop wise slowly and stirred for 10 minutes for eliminating the excess of KMnO₄. The solution was cooled. 20ml of Hydrochloric Acid (HCl) and 60ml of deionized water was added and centrifuged for 7 minutes. The supernatant was decanted away and the residuals were rewashed again with HCl and deionized water for 3 times. The washed GO solution was dried in oven at 90^oC for 24 hours. The GO powder obtained is of greenish-black colour.

2.2 Synthesis of GO-CdS, GO-(Cd $_{0.8}$ -Zn $_{0.2}$)S, GO-TiO $_2$ by Chemical precipitation method:

0.075g of Graphene oxide was dissolved in hot triple distilled water, stirred for 1 hour and added to Cadmium acetate followed by Triethanolamine (TEA), Thiourea and 25% aqueous ammonia. The mixture was then stirred for 30 minutes and to it Mercapthoethanol and Methanol in the ratio 1:1 was added as capping agents. The entire solution was again stirred for 30 minutes and then kept in water bath at 80°C for 2 hours. The obtained precipitate was filtered and dried at room temperature for 24 hours. The GO-CdS powder obtained was dark-yellowish in colour.

For the synthesis of GO-(Cd_{0.8} Zn_{0.2})S, 0.075g of Graphene oxide was dissolved in hot triple distilled water, stirred for 1 hour and added to Cadmium acetate and Zinc acetate in appropriate ratio followed by Triethanolamine (TEA), Thiourea and 25% aqueous ammonia. The same procedure as in the case of GO-CdS was followed afterwards. The GO-(Cd_{0.8} Zn_{0.2})S powder obtained is light-yellowish in colour [12].

For the synthesis of GO-TiO₂, 0.075g of Graphene oxide was dissolved in hot triple distilled water and stirred for 1 hour. It was then added to Titanium Chloride (TiCl₃) solution in 15% HCl followed by deionized water and stirred vigorously for 1.5 to 2 hours. A blue-violet solution was obtained to which Sodium Hydroxide (NaOH) was added drop-wise. The precipitate was filtered and dried at 60° C for 24 hours. The obtained GO-TiO₂ powder was off-white in colour [13].

2.3 Preparation of GO-CdS-(Cd_{0.8}-Zn_{0.2})S, GO-CdS-TiO₂ by Chemical precipitation method:

For the synthesis of GO-CdS-(Cd_{0.8}-Zn_{0.2})S, 0.075g of Graphene oxide was dissolved in hot triple distilled water, stirred for 1 hour and added to Cadmium acetate followed by Triethanolamine (TEA), Thiourea and 25% aqueous ammonia. The mixture was then stirred for 30 minutes and to it Mercapthoethanol and Methanol in the ratio 1:1 was added as capping agents. (Cd_{0.8} Zn_{0.2})S powder prepared separately by Chemical bath precipitation method was taken in the same ratio as that of GO, and mixed with the above solution and further stirred for 30 minutes. The solution was then kept in water bath at 80^oC for 2 hours. The obtained precipitate was filtered and dried at room temperature for 24 hours. The GO-CdS-(Cd_{0.8}-Zn_{0.2})S nanocomposite was bright-yellowish in colour.

For the synthesis of GO-CdS-TiO₂, 0.075g of Graphene oxide was dissolved in hot triple distilled water, stirred for 1 hour and added to Cadmium acetate followed by Triethanolamine (TEA), Thiourea and 25% aqueous ammonia. The mixture was then stirred for 30 minutes and to it Mercapthoethanol and Methanol in the ratio 1:1 was added as capping agents. TiO₂ powder prepared separately by Sol-gel method was taken in the same ratio as that of GO, and mixed with the above solution and further stirred for 30 minutes. The solution was then kept in water bath at 80° C for 2 hours. The obtained precipitate was filtered and dried at room temperature for 24 hours The GO-CdS-TiO₂ nanocomposite was of light-yellow colour.

2.4 Characterization Methods:

2. 4. 1 XRD, SEM and UV-VIS spectral studies:

XRD studies were carried at NIT Raipur using PANalytical 3KW X'pert powder multifunctional X-ray diffractometer, SEM and EDX studies were also conducted at NIT Raipur using ZEISS EVO 18 Scanning Electron Microscope and INCA 250 EDS with X-MAX 20nm detector. Optical absorption spectral studies were performed by using ELICO-SL210 UV-VIS spectrophotometer.

2. 4. 2 Measurement of photocatalytic degradation of Rhodamine B dye:

Photocatalytic degradation of Rhodamine B (RhB) dye solution was carried out in UV-VIS spectrophotometer. 0.03% of RH.B dye solution was taken to which an appropriate quantity of $GO/CdS/(Cd_{0.8} Zn_{0.2})S/TiO_2$ binary and ternary nanocomposites were added and mixture is magnetically stirred for 30 minutes to obtain adsorption-desorption equilibrium. The absorbance of the solution is measured before making light incident on the solution and subsequently after every 30 minutes for 2 hours. The degradation of the dye and change in its absorbance was studied with respect to time. The photocatalytic efficiency of the synthesized nanocomposites was then calculated.

III. RESULTS AND DISCUSSION

3.1 X-ray Diffraction (XRD) studies:

Fig.1 (a) represents the X-ray diffractogram of GO-CdS powder prepared by Chemical bath precipitation and the corresponding data are presented in Table 1 (a). The assignments of the different peaks were made by comparison with JCPDS data and calculation of lattice constants, which showed agreement with reported values.



Fig.1 (a): X-ray diffractogram of GO-CdS powder prepared by Chemical Bath precipitation method.

		o pomaer prep	precipitation method				
20	'd' value (Å)		Relative intensity		(1.1.1)	Lattice constant (Å)	
20 angle	Obs	Rep	Obs	Rep	(ПКІ)	Obs	Rep
26.7301	3.3324	3.3570	100	59	(002) _H CdS	c=6.664	a=4.135 c=6.712
34.0042	2.6343	2.4502	61	25	(102) _H CdS	a=4.301 c=6.664	a=4.135 c=6.712
44.1408	2.0500	2.0580	70	80	(220) _C CdS	a=5.798	a=5.818
52.3604	1.7459	1.7530	63	60	(311)c CdS	a=5.790	a=5.818

Table 1 (a): XRD data of GO-CdS powder prepared by Chemical Bath precipitation method.

The XRD pattern exhibit the prominent broad peaks corresponding to (002), (102) of hexagonal CdS and (220), (311) of cubic crystal lattice structure of CdS. No characteristics peaks corresponding to the (001) plane of GO or (002) plane of Graphene was observed. It is likely that the (001) peak of GO might have overlapped with the prominent peaks of CdS (2θ =26.73°). Also, this may be due to the fact that GO has been reduced to Graphene and the reduced GO sheets might have been exfoliated by the deposition of the CdS on their surface thereby forming loosely packed individual sheets [14].



Fig.1 (b): X-ray diffractogram of GO-(Cd_{0.8}Zn_{0.2})S powder prepared by Chemical Bath precipitation method.

20 angla	'd' value (Å)		Relative intensity		(b bl)	Lattice constant (Å)	
20 angle	Obs	Rep	Obs	Rep	(IIKI)	Obs	Rep
25.9664	3.3037	3.357	100	59	(002) _H CdS	c=6.607	a=4.135
					()11		c=6.712
33 3215	2 6867	2.66	63	2	$(107)_{1011}$ 7nS	a=3.83	a=3.820
55.5215	2.0007	2.00	05	2	(107)I0H ZIIS	c=31.83	c=31.20
44 0620	2 0535	2.058	66	80	(220) _C CdS	a=5.808	a=5.818
++.0020	2.0555	2.050	00	00			
47 4406	1.0145	1.011	<i>C</i> 1	74	(110) 79	a=3.83	a=3.820
47.4490	1.9145	1.911	04	/4	$(110)_{10H}$ ZHS		c=6.260
50.0049	1 7004 1 7012	65	17	(200) C4C	- 1122	a=4.135	
50.9948	1./894	1.7915	65	17	$(200)_{\rm H}$ CdS	a=4.152	c=6.712

 Table 1 (b): XRD data of GO-(Cd_{0.8}Zn_{0.2})S powder prepared by Chemical Bath precipitation method.

Fig.1 (b) represents the X-ray diffractogram of GO-(Cd_{0.8}Zn_{0.2})S powder prepared by Chemical bath precipitation and the corresponding data are represented in Table 1 (b). The diffraction peak corresponding to the maximum intensity is $(002)_{\rm H}$ plane of hexagonal CdS (same as that observed in GO-CdS). (220)_C plane of cubic CdS is observed in this case too. In addition the (200) plane of hexagonal CdS has been obtained which was absent in GO-CdS. (107) and (110) hexagonal plane of ZnS are also observed.



 $Fig. 1 \ (c): X-ray \ diffractogram \ of \ GO-CdS-(Cd_{0.8}Zn_{0.2})S \ powder \ prepared \ by \ Chemical \ Bath \ precipitation \ method.$

20 angla	'd' va	lue (Å)	Relative intensity		(h]-1)	Lattice constant (Å)	
20 angle	Obs	Rep	Obs	Rep	(IIKI)	Obs	Rep
26.8351	3.3196	3.357	100	59	(002) _H CdS	c=6.639	a=4.135 c=6.712
43.9570	2.0582	2.058	56	80	(220) _C CdS	a=5.821	a=5.818
47.8698	1.8987	1.8979	50	42	(103) _H CdS	a=3.695 c=6.639	a=4.135 c=6.712
51.5200	1.7724	1.764	47	52	(103) _{2H} ZnS	a=3.83 c=6.249	a=3.82 c=6.260
54.0410	1.6955	1.679	43	4	(004) _C CdS	c=6.782	a=4.135 c=6.712

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Fig.1 (c) represents the X-ray diffractogram of GO-CdS-(Cd_{0.8}Zn_{0.2})S powder prepared by Chemical bath precipitation and the corresponding data are represented in Table 1 (c). The XRD pattern display the prominent broad peaks of CdS at (002)_H and (220)_C planes (same as that observed in GO-CdS). In addition to these planes (103) and (004) of hexagonal CdS along with (103) plane of hexagonal ZnS was observed.

Fig.1 (d) represents the X-ray diffractogram of GO-CdS-TiO₂ powder and the corresponding data are presented in Table 1 (d). The XRD pattern exhibit the prominent broad peaks of CdS at $(220)_{C}$ planes (same as that observed in GO-CdS). It also shows the additional planes of (222), (400), (331) and (420) of the cubic crystal lattice structure and (103) plane of hexagonal structure of CdS. A mixture of anatase and rutile phases of TiO₂ with (101), (004), (211) of anatase structure and (110) and (301) of rutile structure is seen.



Fig.1 (d): X-ray diffractogram of GO-CdS-TiO2 powder prepared by Chemical Bath precipitation method.

Tabl	Cable 1 (d): XRD data of GO-CdS-TiO2 powder prepared by Chemical Bath precipitation method.							
20 angla		'd' va	lue (Å)	Relative	e intensity		Lattice co	nstant (Å)
	20 angle	Obs	Rep	Obs	Rep	(IKI)	Obs	Rep
	25.5746	3.4802	3.5200	100	100	(101) _A TiO ₂	a=3.743 c=9.452	a=3.785 c=9.513
	27.7017	3.2176	3.2470	78	100	(110) _R TiO ₂	a=4.550	a=4.593 c=2.959
	38.0484	2.3631	2.3720	47	20	(004) _A TiO ₂	c=9.452	a=3.785 c=9.513
	44.3246	2.0419	2.0580	50	80	(220) _C CdS	a=5.775	a=5.818
	48.2637	1.8841	1.8979	54	42	(103) _H CdS	a=4.226 c=6.603	a=4.1354 c=6.7120
	54.3037	1.6879	1.6800	41	10	(222) _C CdS	a=5.847	a=5.818
	55.2490	1.6612	1.6665	44	20	(211) _A TiO ₂	a=3.773 c=9.452	a=3.785 c=9.513
	62.9696	1.4749	1.4530	34	20	(400) _C CdS	a=5.899	a=5.818
	69.1671	1.3571	1.3598	32	20	(301) _R TiO ₂	a=4.581 c=2.959	a=4.593 c=2.959
	70.5327	1.3370	1.337	32	30	(311) _C CdS	a=5.815	a=5.818
	75.3121	1.2649	1.298	33	10	(420) _C CdS	a=5.804	a=5.818

le	1 (d):	: XRD	data o	f GO-	CdS-TiO ₂	powder	prepared b	v Che	emical Batł	1 precipitatio	on method.
IC.	1 (u)		uata 0	100-	Cu0-1102	powder	pi cpai cu i	y en	innear Dati	i pi ccipitati	m memou.

Table 2 shows the particle size of the synthesized materials of the binary and ternary nanocomposites

Table 2: Particle size of nanocomposites

S No.	Sample	Particle size(nm)
1	GO-CdS	7.95
2	$GO-(Cd_{0.8}Zn_{0.2})S$	8.17
3	GO-CdS-(Cd _{0.8} Zn _{0.2})S	8.01
4	GO-CdS-TiO ₂	10.88

3.2 Scanning Electron Microscopy (SEM) studies:



Fig.2 (a) SEM micrograph of GO-CdS

Fig 2 (b) SEM micrograph of GO-(Cd_{0.8}Zn_{0.2})S

Fig 2 (a) and 2 (b) shows the SEM micrographs of GO-CdS and GO-($Cd_{0.8}Zn_{0.2}$)S nanocomposites respectively at a magnification of 50 KX. Non-uniform distribution of spherical particles forming clusters on the surface of GO sheets is observed in both cases.



Fig 2 (c) SEM micrograph of GO-CdS-(Cd_{0.8}Zn_{0.2})S nanocomposite

Fig 2 (d) SEM micrograph of GO-CdS-TiO₂ nanocomposite

Whereas fig 2 (c) and 2 (d) shows the SEM images of ternary nanocomposites of GO-CdS- ($Cd_{0.8}Zn_{0.2}$)S and GO-CdS-TiO₂ at the magnification of 50 KX. Similar morphology is observed in both the cases. In the GO-CdS-TiO₂ composites the spherical nature of the particles is less evident. Some irregular shaped particles agglomerating on the surface of GO can be seen.

3.3 Energy Dispersive X-ray Analysis (EDX):

Element	Weight%	Atomic%
C K	23.38	55.21
O K	10.39	18.42
S K	15.30	13.53
Cd L	50.93	12.85

Table 3 (a) EDX data of GO-CdS

Element	Weight%	Atomic%
C K	28.73	60.50
O K	10.35	16.36
S K	16.04	12.66
Zn K	2.30	0.89
Cd L	42.58	9.58

Table 3 (b) EDX data of GO-(Cd_{0.8}Zn_{0.2})S



Fig. 3 (a) EDX Spectra of GO-CdS



Fig. 3 (b) EDX Spectra of GO-(Cd_{0.8}Zn_{0.2})S

Fig. 3 (a) and 3 (b) shows the EDX spectra of the binary nanocomposites of GO-CdS and GO-($Cd_{0.8}Zn_{0.2}$)S. The weight% and atomic% in both cases are presented in tables 3 (a) and 3 (b) respectively. The presence of C, O, S, Cd with excess Carbon is observed in both the cases. A small percentage of Zn in addition to the other elements has been observed in GO-($Cd_{0.8}Zn_{0.2}$)S. No impurities have been detected in both the materials.

Element	Weight%	Atomic%
C K	38.55	68.30
O K	12.43	16.54
S K	12.23	8.12
Zn K	0.59	0.19
Cd L	36.20	6.85

Table 3 (c) EDX data of GO-CdS-(Cd_{0.8}Zn_{0.2})S

Element	Weight%	Atomic%
C K	42.15	61.43
O K	28.04	30.68
S K	5.87	3.20
Ti K	4.55	1.66
Cd L	19.39	3.02

Table 3 (d) EDX data of GO- CdS-TiO₂



Fig. 3 (c) EDX Spectra of GO-CdS-(Cd_{0.8}Zn_{0.2})S



Fig. 3 (d) EDX Spectra of GO-CdS-TiO₂

Fig. 3 (c) and 3 (d), shows the EDX spectra of the ternary nanocomposites of GO-CdS-(Cd_{0.8}Zn_{0.2})S and GO-(Cd_{0.8}Zn_{0.2})S-TiO₂ respectively. Excess Carbon along with Cd, Zn, S, O has been detected in GO-CdS-(Cd_{0.8}Zn_{0.2})S. A similar elemental composition is observed in GO-(Cd_{0.8}Zn_{0.2})S-TiO₂ with Zn being replaced by Ti.

3.4 Photocatalytic degradation of Rhodamine B Dye:

Photocatalytic degradation of Rhodamine B dye was carried out with single materials as well as binary and ternary composites as catalysts. Rhodamine B dye has a prominent absorption peak at 554nm and the absorbance at this wavelength decrease with decrease in concentration. [4] Figs 5 (a), (b) and (c) show the absorbance vs wavelength plot of degraded dye solution at different irradiation time of CdS, $(Cd_{0.8}Zn_{0.2})S$ and TiO₂ nanoparticles. It can be observed from the figures that the absorbance of the solution decreases with increase in irradiation time. However, the efficiency of photocatalytic degradation is very less in the case of single materials.



Figs 5 (d), (e) and (f) shows the absorbance vs wavelength plot of degraded dye solution for GO-CdS, GO-($Cd_{0.8}Zn_{0.2}$)S and GO-TiO₂ respectively. The decrease in absorbance with irradiation time is more prominent in this case suggesting better photocatalytic efficiency for binary composites. This clearly indicates that introduction of CdS, ($Cd_{0.8}Zn_{0.2}$)S and TiO₂ in GO matrix improves the photocatalytic activity as a result of the high surface to volume ratio and specific surface area of GO sheets that could disperse CdS, ($Cd_{0.8}Zn_{0.2}$)S and TiO₂ nanoparticles and allow for greater photon absorption and correspondingly higher photocatalytic activity [5, 15].



In, fig 5 (g) and (h) shows the absorbance vs wavelength plot of $GO-CdS-(Cd_{0.8}Zn_{0.2})S$, $GO-CdS-TiO_2$ ternary nanocomposites. A remarkable decrease in absorption with irradiation time is observed for the ternary nanocomposites. Figs 6 (a), (b) and (c) shows the concentration vs time plot of Rh. B dye with single material, binary and ternary nanocomposites as catalysts.



Fig 5 (g) Optical Absorption Spectra of degraded dye solution after different time intervals with GO-CdS-(Cd_{0.8}Zn_{0.2})S as catalyst

Fig 5 (h) Optical Absorption Spectra of degraded dye solution after different time intervals with GO-CdS-**TiO₂** as catalyst

(1)

Figs 6 (a), (b) and (c) shows the concentration vs time plot of Rh. B dye with single material, binary and ternary nanocomposites as catalysts.



The photocatalytic efficiency was calculated using the relation

[C_o-C/C_o] x 100

Where C_0 is the initial concentration and C is the concentration after different times and the corresponding values are summarized in table 4. It can be concluded from the table that photocatalytic performance was best in case of ternary nanocomposites suggesting that these nanocomposites can be utilized for water treatment with specific application to removal of dyes from waste water.

Table 4: Photocatalytic efficiency % of the synthesized nanomaterials

S.No.	Sample	%Efficiency
1	CdS	10
2	(Cd _{0.8} Zn _{0.2})S	12
3	TiO ₂	11
4	GO- CdS	22
5	GO-(Cd _{0.8} Zn _{0.2})S	20
6	GO-TiO ₂	21
7	GO-CdS-(Cd _{0.8} Zn _{0.2})S	42
8	GO-CdS- TiO ₂	60

IV. CONCLUSION

Successful synthesis of GO-CdS, GO-(Cd_{0.8} Zn_{0.2})S, GO-TiO₂ binary composites and GO-CdS-(Cd_{0.8} Zn_{0.2})S, GO-CdS-TiO₂ ternary nanocomposites was carried out by chemical bath precipitation method. The photocatalytic properties of the synthesized composites were investigated by the measurement of photocatalytic degradation of Rhodamine B dye and the photocatalytic efficiency was calculated. Ternary nanocomposites were found to show better photocatalytic efficiency in comparison to binary and single materials. Structural properties studied using XRD measurements showed planes of CdS and ZnS in binary nanocomposites and planes corresponding to CdS, ZnS and TiO₂ in ternary nanocomposites. Morphology of the nanocomposites was observed from SEM micrographs which showed a non-uniform distribution of spherical particles forming clusters. The elemental analysis was done using EDX spectra which confirmed the presence of C, O, S, Cd, Zn and Ti with excess Carbon.

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