Research Article

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Transition Metal Doping for Sustainable Catalysis: Enhancing the Structural and Catalytic Properties of Graphene Oxide Integrated BSA-stabilized CdSe Nanoparticles

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ABSTRACT

The research work presented here describes the influence of transition metal (Fe, Ni, Co) doping in graphene oxide (GO) integrated biomolecule stabilized CdSe nanoparticles on the structural and catalytic properties. The CdSe and doped CdSe were synthesised by simple chemical co-precipitation method. These molecules were stabilized by bovine serum albumin (BSA) in order to overcome the agglomeration and achieve water dispersibility. The synthesized molecules were then made into composites by integrating with graphene oxide (GO) and characterized by powder X-ray diffraction (PXRD), UV-Vis spectroscopy, Fourier transform Infrared spectroscopy (FTIR), scanning electron microscopy (SEM). Further, the synthesized materials were tested for catalytic reduction of *p*-nitro aniline into *p*-phenylenediamine under the optimized conditions. These results indicated that doping of cobalt has notable influence on the structural and catalytic properties. About 98.73% of conversion was achieved in the reduction reaction on using GO/Cobalt doped BSA-CdSe as the catalyst.

Keywords: CdSe nanoparticles; Transition metal doping; Bovine serum albumin; Graphene oxide composites; Catalytic reduction.

1. INTRODUCTION

Graphene oxide (GO) and its composites have emerged as promising materials in various fields due to their unique structural, electrical, and chemical properties (Mushahary *et al.* 2024). The ability to integrate GO with other functional materials, such as nanoparticles, has opened new avenues for enhancing its applications in areas like catalysis (Gao *et al.* 2024), energy storage(Kalyan *et al.* 2022; Mousavi *et al.* 2023), and sensing (Matthew *et al.* 2024). In particular, the combination of GO with semiconductor nanoparticles like cadmium selenide (CdSe) has gained significant attention because of the resulting enhancement in properties that can be tailored for specific applications(Huo *et al.* 2017; Xie *et al.* 2016). CdSe nanoparticles are well-known for their size-dependent optical and electronic properties, making them ideal candidates for applications in photovoltaics, optoelectronics, and photocatalysis(Colvin *et al.* 1994; Dayal *et al.* 2010; Hu *et al.* 2009; Min *et al.* 2007; Ullah *et al.* 2014). However, the stability and performance of these nanoparticles can be significantly influenced by their environment. To address this, bovine serum albumin (BSA) has been employed as a stabilizing agent to prevent the aggregation of CdSe nanoparticles, ensuring their uniform dispersion and stability in aqueous solutions(Ahmed *et al.* 2014; Singh *et al.* 2016). The incorporation of BSA not only stabilizes the nanoparticles but also introduces functional groups that can interact with GO, leading to the formation of stable nanocomposites(Liu *et al.* 2022; Pérez-Piñeiro *et al.* 2023). Transition metal doping is a well-established strategy for tuning the properties of semiconductors (Zheng *et al.* 2025). By introducing transition metals such as iron (Fe), cobalt (Co), and nickel (Ni) into the CdSe lattice, it is possible to modify the electronic, magnetic, and catalytic properties of the material. These modifications can result in enhanced performance in various applications, particularly in catalysis, where the doped materials can offer improved efficiency and selectivity. The doping of transition metals into CdSe

nanoparticles, when combined with GO, could lead to the development of advanced nanocomposites with superior structural and catalytic properties. The unique two-dimensional structure of graphene oxide offers a large surface area, which is ideal for supporting and dispersing nanoparticles. This makes GO an excellent candidate for creating nanocomposites with enhanced physical and chemical properties. The interaction between GO and CdSe nanoparticles can lead to synergistic effects. The choice of transition metals like Fe, Co, and Ni for doping is strategic. Each of these metals has distinct electronic configurations and catalytic properties, which can induce different effects when incorporated into the CdSe lattice. For instance, Fe doping can introduce magnetic properties, Co can enhance electronic conductivity, and Ni can improve the catalytic activity for specific reactions(Almontasser and Parveen, 2022; Parastar *et al.* 2024; Yang *et al.* 2023). By systematically studying these effects, we can gain a deeper understanding of how to tailor nanocomposites for specific catalytic applications. Moreover, the integration of these doped nanoparticles with GO not only stabilizes the nanocomposites but also facilitates electron transfer during catalytic reactions. This can significantly enhance the efficiency of catalytic processes, making these materials highly desirable for applications such as pollutant degradation, hydrogen production, and other environmentally relevant reactions.

In this research, we explore the influence of transition metal doping on the structural and catalytic properties of GO-integrated BSA-stabilized CdSe nanoparticles. The study focuses on the synthesis of GO/BSA-CdSe composites doped with Fe, Co, and Ni, and the subsequent characterization of these nanohybrids using various analytical techniques. The structural properties of the synthesized materials are investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR), while their catalytic performance was evaluated in the reduction of *p*-nitroaniline, a widely used model reaction for evaluating the catalytic activity of nanomaterials. This study aims to provide insights into how transition metal doping can influence the structural integrity and catalytic efficiency of GO-integrated CdSe nanoparticles. The findings could have significant implications for the design and development of nanocomposites for various catalytic applications, particularly in environmental remediation and energy conversion. By exploring how doping elements affect the properties of the nanocomposites, this research advances the broader field of nanotechnology, where the capability to precisely modify material characteristics is essential.

2. MATERIALS AND METHODS

2.1 Materials

Graphite powder, potassium permanganate ($KMnO_4$), selenium powder, sodiumborohydride, cadmium acetate, iron(III) chloride, nickel chloride, cobalt chloride

2.2 Preparation of Graphene Oxide (GO) by modified Hummers Method

Graphene oxide was prepared using modified Hummer's method (Varaprasad *et al.* 2017). The expanded graphite powder (18 g) was mixed with concentrated H_2SO_4 (480 mL). $KMnO_4$ (62 g) was added slowly by magnetic stirring under ice-cold conditions. The temperature of the mixture was maintained just above 20°C. The mixture was stirred at 35°C for 2 hours and deionized water (920 mL) was added. Approximately, in 1 hour, the reaction was terminated by the addition of a large amount of deionized water (2.8 L) and 30% H_2O_2 solution (50 mL), causing violent effervescence and an increase in temperature to 100°C. The bright yellow coloured suspension was washed with 1:10 HCl solution (5 L) in order to remove metal ions using filter paper and funnel. The paste collected from the filter paper was dried at 60°C, until it became an agglomeration. The agglomeration was dispersed into deionized water in static state for ~2-3 hours and slightly stirred with a glass rod. The suspension was washed with deionized water for ~5-7 times for two days using filter paper and funnel, until the pH reached nearly 7. The paste collected on the filter paper was dispersed into water by ultrasonication. The obtained brown dispersion was subjected to 30 min centrifugation at 4000 rpm to remove any unexfoliated GO using a centrifuge. The GO platelets were air dried at 60°C.

2.3 Synthesis and Characterization of GO/BSA-CdSe, GO/BSA-Fe:CdSe, GO/BSA-Co:CdSe, GO/BSA-Ni:CdSe Nanohybrid Composites

The CdSe quantum dots (QD) were prepared by chemical co-precipitation method which involves the mixing of selenium powder (0.32 mmol) with deionized water (20 mL) in a three-necked flask. Sodium borohydride (0.79 mmol) was carefully added to this mixture under nitrogen gas. The selenium solution turned to a light pink solution after 2 hours. In another beaker, cadmium acetate (4 g) was dissolved in deionized water (250 mL) along with bovine serum albumin (0.5%w/w). Then these two solutions were stirred at 90 °C for 6 hrs to get a red colour solution. The pH of the mixture was kept at 6 by adding KOH. At this point, 0.01-0.2 mL of 0.1 M of the metal solution was added dropwise. The obtained quantum dots were then precipitated and washed several times by using 2-propanol and then purified, dried at room temperature in vacuum overnight. Above prepared QDs were dispersed during GO synthesis to obtain QDs integrated GO as nanocomposites.

2.4 Characterization Methods

The synthesized samples were well dried for characterization purpose. The powder X-ray diffraction

(XRD) studies were performed on Bruker D8 Discover A25 Germany with a scan rate of 5° per minute. The field emission scanning electron microscope images were recorded on JEOL SEM IT 300 with EDS (Oxford instruments, UK). The Fourier transform infrared spectroscopy studies were recorded on Nicolet iS5 from HERMO Electron Scientific Instruments LLC, USA. The UV-Visible spectra studies were conducted on Perkin Elmer Lambda 20 UV-Vis spectrophotometer.

2.5 Catalytic Reduction

About 0.6906 g of *p*-nitroaniline and 0.189 g of $NaBH_4$ were dissolved in 50 mL of deionized water to make homogeneous solutions. Subsequently, 400 µL of resulting solution was mixed in 3200 µL of deionized water. From this solution, 1500 µL was taken into a cuvette and diluted with 1500 µL of deionized water. To this diluted solution, 0.05 g of the prepared nanocomposites were added as catalyst. The catalytic activity of the nanocomposite was monitored by UV- Vis spectrophotometer.

3. RESULTS AND DISCUSSION

The optical absorption spectra of the synthesized nanocomposites of GO/BSA-CdSe and transition metal doped CdSe samples are presented in Fig. 1. All the samples exhibited absorption in the visible region with a wavelength range 440 nm – 460 nm. Also, the intensities of the spectra were almost similar. Among the three dopants, cobalt-doped composite showed a very slight shift. This may be attributed to the introduction of unoccupied 3d electrons in the crystal structure of CdSe (Šutka *et al.* 2016). However, the doping of Fe and Ni did not show any specific change in the optical spectra. The broad UV spectra for the bulk as well as the doped GO/BSA-CdSe composite indicate that the prepared composites are in nanometer range as observed earlier by (Saravanan *et al.* 2011).

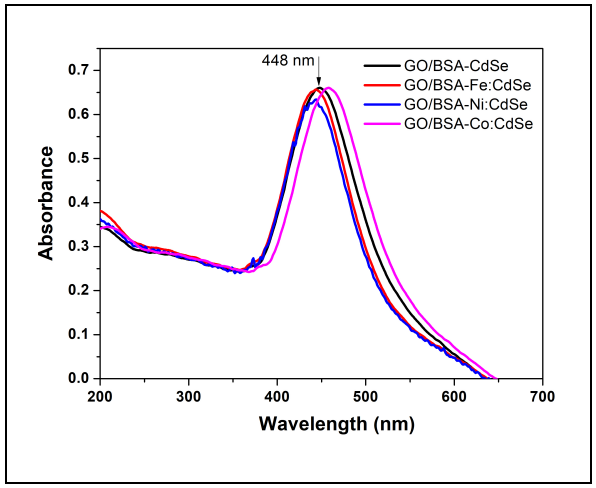


Fig. 1: UV-Vis spectra of synthesized GO integrated metal doped CdSe nanocomposites

The FTIR spectrum of transition metal-doped GO/BSA-CdSe were recorded and are presented in Fig. 2. The peaks at 3425 $cm^{-1}$and 1628 cm^{-1}represent the O-H and C=O stretching vibrations. This maybe due to presence of water molecules in GO or introduction of the same during the process. The influence of BSA has been discussed earlier (Mullamuri *et al.* 2017). The peaks observed at 1055 cm^{-1} and 657 cm^{-1} were attributed to the presence of CdSe. Interestingly, no change in peak position was observed even in the doped composites, which attests that the dopants were included in the crystal lattice without disturbing the CdSe network. These results confirmed the functionalization of CdSe, which is due to amide and hydroxyl groups in BSA. However, further investigation is required to understand the mechanism.

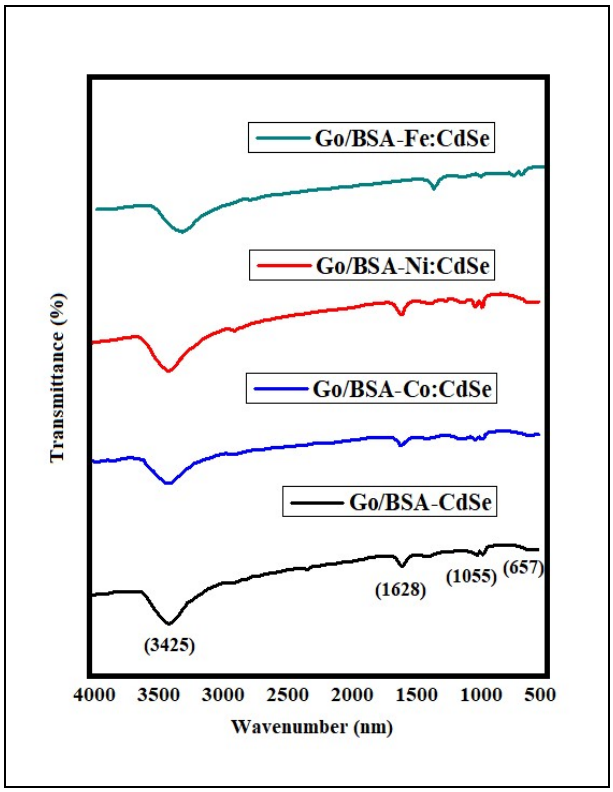


Fig. 2: FT IR spectra of nanocomposites

The XRD patterns of synthesized nanocomposites are presented in Fig. 3. The formation of BSA-CdSe patterns were in accordance with the JCPDS CAS no: 08-0459. These patterns include the major peaks of the graphene oxide which further attests the formation of GO/BSA-CdSe nanocomposites. These direction patterns were observed at 2θ values 13.84, 27.38, 29.82, 41.36, 45.50, 51.74, 61.49, 65.30. These values correspond to hexagonal structure of CdSe reported in the literature(Jain and Verma, 2015). Further, it was observed from the figure that the doping of metal ions in the crystalline structure of CdSe may lead to a small shift in the peak positions. This shift can be attributed to the size of the metal ion doped(Mohayman *et al*. 2024; Phuc and

Tung, 2019). The ionic radii of iron, cobalt and nickel are 64 pm, 79 pm and 83 pm, respectively while the ionic radius of Cd is 109 pm, which is greater than the ionic radii of the dopants. Hence, it can be noticed that the shift was towards higher angles with respect to the reflection planes. The average crystalline size of the nanocomposites was calculated by Debye-Sherrer formula. Sizes of 8.09 nm, 7.98 nm, 8.22 nm and 7.85 nm for GO/BSA-CdSe, GO/BSA-Fe:CdSe, GO/BSA-Ni:CdSe and GO/BSA-Co:CdSe, respectively were reported.

The SEM micrographs are presented in Fig. 4 (a1-a4). These images show that the nanomaterials are clearly agglomerated over the GO sheets. Further, the doped nanomaterials were found in different shapes which may be attributed to Oswald ripening (Kumar *et al.* 2012; Li *et al.* 2006). The EDS spectra are presented in Fig. 4 (b1-b4). The data correlate with the elements in the composition, confirming the homogeneous and uniform distribution of the composites.

3.1 Catalytic Reduction of *p*-nitroaniline

The catalytic application of synthesized nanomaterials is shown in scheme 1. The reduction takes place in the presence of sodium borohydrate as the reducing agent and the GO integrated BSA stabilized nanomaterials as the catalyst. The catalytic performance was monitored by UV-Visible spectroscopy in real time. Earlier, our group has shown (Rapaka *et al.* 2024)that the reduction reaction was not successful even after a reaction time of 24 hours in the absence of catalyst However, in the presence of GO/BSA-CdSe, GO/BSA-Fe:CdSe, GO/BSA-Co:CdSe and GO/BSA-Ni:CdSe nanocomposites the reduction process was complete. Particularly, GO/BSA-Co:CdSe aided the highest yield of *p*-phenylenediamine in 110 mins. Fig. 5 clearly shows that the absorption peak of *p*-nitroaninline noticed at 381 nm was subsequently shifted to 303 nm after complete reduction. Thus, the synthesized nanocomposites were efficient catalysts for the reduction of *p*-nitroaninline. Also, the doping of transition metal increases the efficiency of the catalytic behaviour of the GO/BSA-CdSe nanocomposite. The conversion percentage of *p*-nitroaninline to *p*-phenylenediamine was approximately 87.73% for GO/BSA-CdSe, 89.08% for GO/BSA-Fe:CdSe, 91.62% GO/BSA-Ni:CdSe and 98.73% for GO/BSA-Co:CdSe in 110 min. However, further studies are required to understand the mechanism, influence of doping and efficiency of the catalytic reduction.

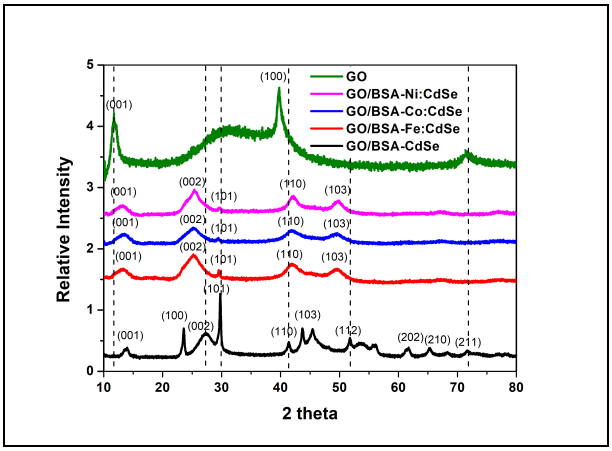
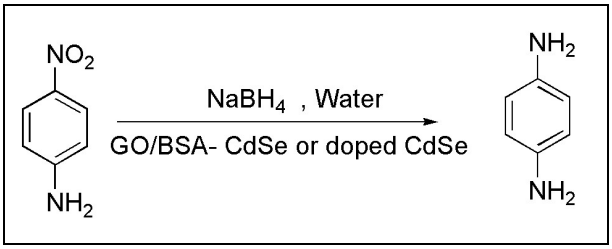
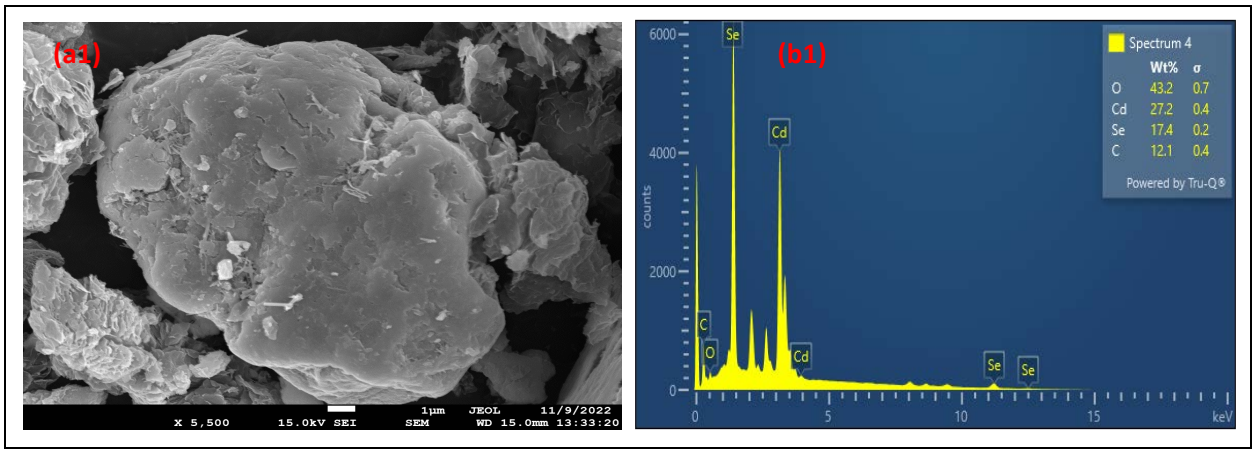


Fig. 3: X-ray Diffraction patterns of synthesized GO/BSA-CdSe and transition metal doped CdSe nanocomposites



Scheme 1: Catalytic reduction of p-nitro aniline into para phenylene diamine employing GO/BSA-CdSe and GO/BSA-Fe:CdSe; GO/BSA-Ni:CdSe; and GO/BSA-Co:CdSe



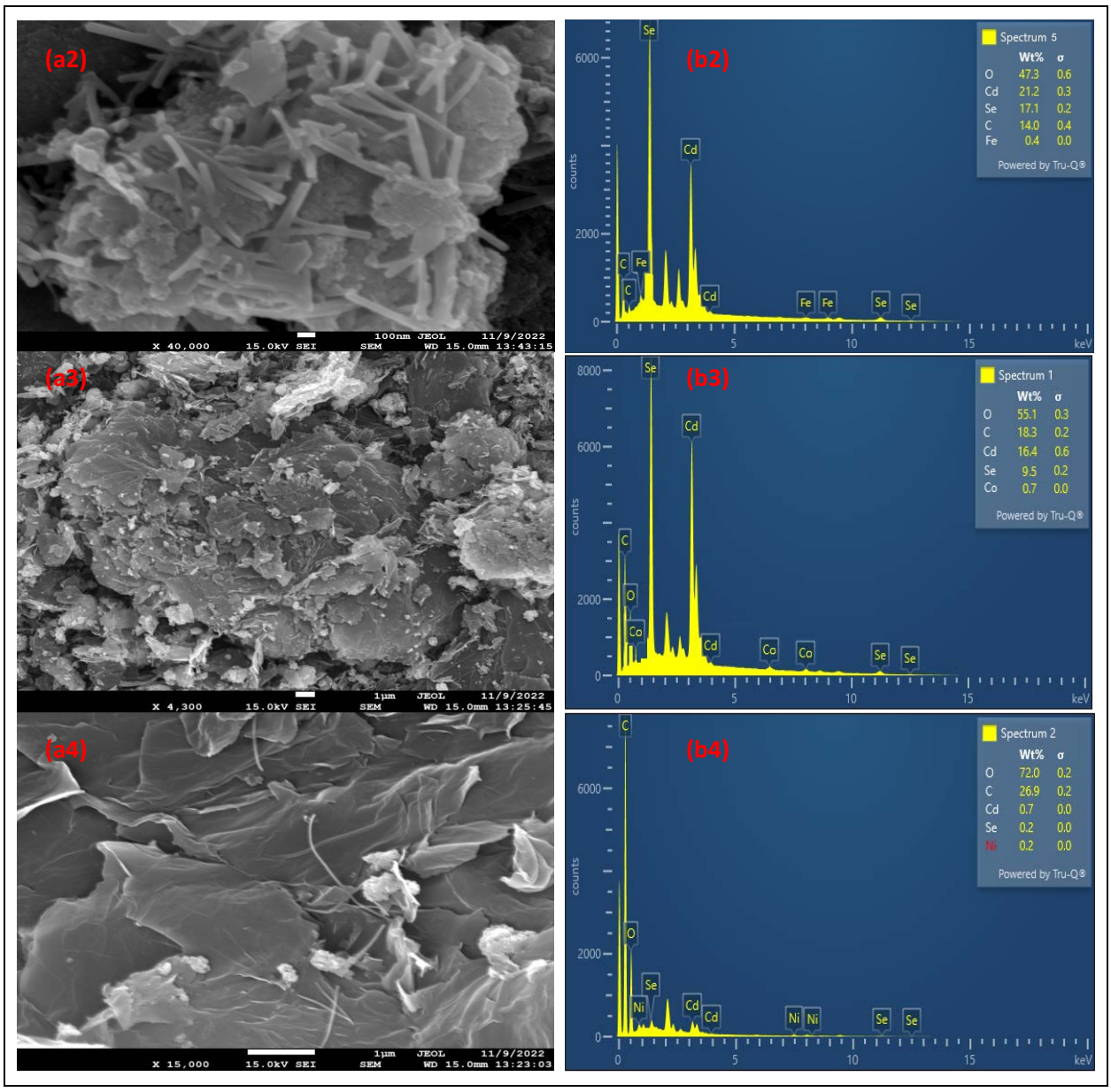
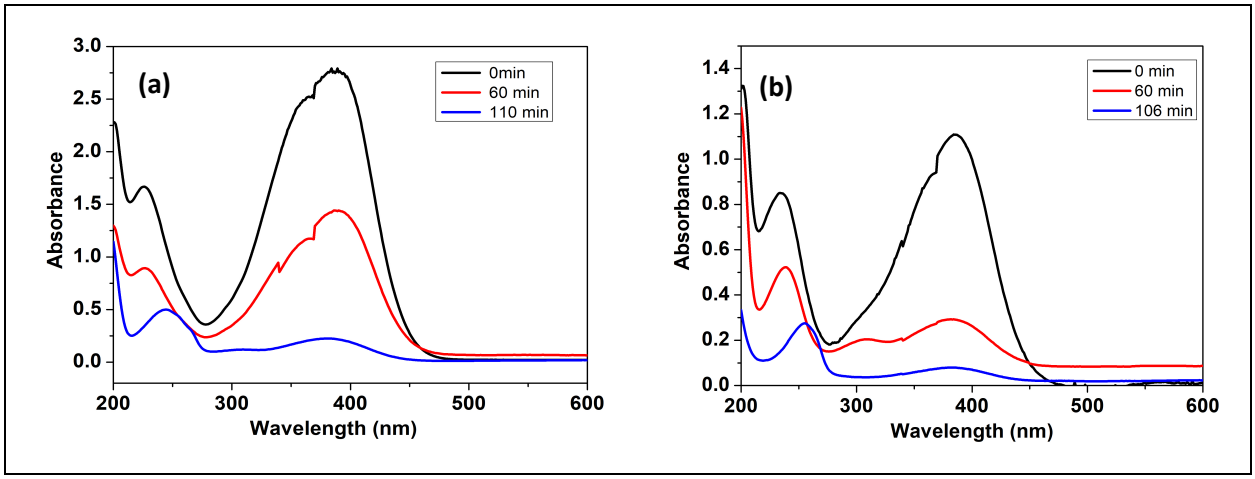


Fig. 4: SEM images (a1-a4) and EDS spectra (b1-b4) of synthesized GO integrated metal-doped CdSe nanocomposites



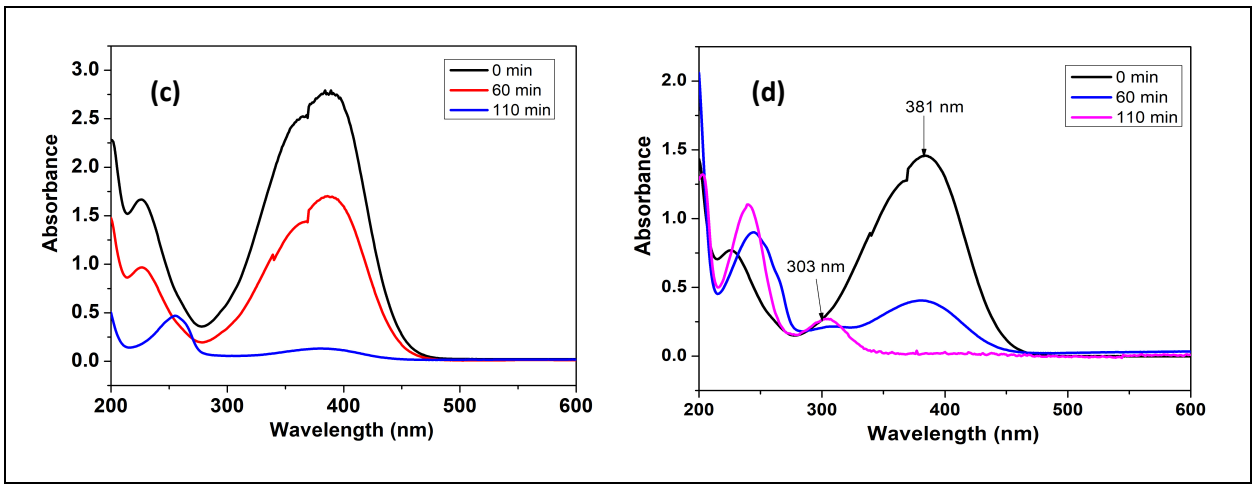


Fig. 5: Catalytic reduction of *p*-nitroaniline into phenylene diamine in presence of $NaBH_4$ (a) GO/BSA-CdSe; (b) GO/BSA-Fe:CdSe; (c) GO/BSA-Ni:CdSe; (d) GO/BSA-Co:CdSe

4. CONCLUSION

In the present investigation, the transition metal (Fe, Co, Ni) doped GO integrated BSA stabilised CdSe nanoparticles were successfully synthesized by chemical co-precipitation method. The developed materials were characterized by UV, XRD, SEM-EDS and FTIR. The XRD studies indicated that there was a small shift observed in the doped CdSe composites, confirming successful doping in the lattice structure of crystalline CdSe. Further, doping influenced the absorption spectrum of CdSe which can be attributed to the introduction of unoccupied 3d electrons in the crystal structure of CdSe. The FTIR spectra revealed that the functional groups in BSA were responsible for the stabilization of CdSe and that doping doesn't influence the crystal lattice of CdSe nanoparticles. The catalytic performance of the synthesized nanocomposites evaluated through the reduction of *p*-nitroaniline to *p*-phenylenediamine revealed that the cobalt-doped GO/BSA-CdSe nanomaterial achieves a 98.73% conversion within 110 minutes.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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