

# Synthesis of Graphene oxide supported with Bimetallic nanoparticles and its Application

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## **ABSTRACT:**

In this study, a very simple and highly effective mechanochemical preparation method was developed for the preparation of Ni nanoparticles supported graphene oxide (GO). The developed method is not only very simple and efficient, but also, the morphology of Ni/GO nanocomposites can be tuned by simply varying the metal loading. The nanoparticle has an immense assortment of prospective applications in biomedical, optical, and electronic fields. Nanoparticles are of great technological fascination as they are effectively an aqueduct between bulk substances and atomic or molecular structures. The properties of matter change as their size approaches to nanoscale and as the atomic percentage at the material surface becomes significant. For bulk materials larger than one micrometre in size, the rate of particles at the surface is minute relative to the total number of particles of the material. The absorbing and occasionally unpredicted properties of nanoparticles are not partly due to the characteristic of the material surface dominating the properties instead of the bulk properties. In this study, two different graphene oxide supported by two mono Ni Mn and bimetallic Ni/Mn nanoparticles catalyst synthesized. The size and shape of the products were characterised by scanning electron microscopy (SEM) and X-ray diffraction spectroscopy (XRD). Results proved that the newly developed graphene oxide carried nickel-manganese nanoparticles catalysts can be more efficient to reductive, oxidative and environmentally important organic pollutants.

**KEYWORDS:** GO, Ni, Mn, UV, SEM, XRD Rhodamine, nanoparticles.

## **1. INTRODUCTION:**

Nanotechnology is a size related to physico- chemical properties differing significantly from larger matter that can rapidly be growing by fabrication nanoproducts and nanoparticles (NPs). In nanotechnology, a particle defined as a small object that behaves as a whole unit with respect to its transport and properties. Nanoparticle research is currently an area of intense scientific interest due to a wide variety of potential applications in biomedical, optical and electronic fields<sup>1-4</sup>. These nanoparticles can be monometallic or bimetallic. Bimetallic nanoparticles are considered superior to monometallic as they have a synergistic effect of both the metals used for their synthesis. Due to their diverse properties, they are used in widespread field such as catalysis, electrochemistry, magnetism, optics, and biomedicine. These applications often highly depend on the structure like shape, size, composition, and surface chemistry of the Nanoparticles<sup>5</sup>.

The nanoparticles are particles which range from size of 1nm to 10 nm having specific physical and chemical properties that are intermediate between those of the bulk metals<sup>6-9</sup>.

Metal oxides play a very important role in many areas of chemistry, physics and materials science. The metal elements can form a large diversity of oxide compounds. These can adopt a vast number of structural geometries with an electronic structure that can exhibit metallic, semiconductor or insulator character. However, the growing importance of surface free energy and stress with decreasing particle size must be considered, changes in thermodynamic stability associated with size can induce modification of cell parameters and/or structural transformations and in extreme cases the nanoparticle can disappear due to interactions with its surrounding environment and a high surface free energy. This review article is focused on the description of metal oxide single crystalline nanostructures used for gas sensing. Metal oxide nano-wires are crystalline structures with precise chemical composition, surface terminations, and dislocation-defect free. Their nanosized dimension generate properties that can be significantly different from their coarse-grained polycrystalline counterpart. Surface effects appear due to magnification in the specific surface of nanostructures, leading to an enhancement of the properties, such as catalytic activity or surface adsorption. High degree of crystalline and atomic sharp terminations makes them

very promising for better understanding of sensing principles and for the development of a new generation of gas sensors.

Monometallic nanoparticles (MNPs), as the name suggests, consist of only single metal. The constituted metal atom determines the properties of these nanoparticles. Monometallic nanoparticles are of different types depending upon the type of metal atom present such as magnetic, metallic and transition metal nanoparticles, etc. They can be prepared by different routes but the most important is the chemical method. Their structure can be stabilized using various functional groups. Last few decades have marked the greater interest in the field of metallic nanoparticles due to their enhanced physical and chemical properties<sup>10-12</sup>.

Metal nanoparticles catalysts composed of two (or more) different metal elements are of interest from both technological and scientific views for improving the catalyst quality or properties<sup>13</sup>. In fact, bimetallic (or multimetallic) catalysts have long been valuable for in-depth investigations of the relationship between catalytic activity and catalyst particle structure. Bimetallic nanoparticles (NPs) are an important class of catalysts. Among these types, bimetallic alloy NPs are very important nanomaterials because of their applications in a large variety of catalytic reactions, including catalytic alcohol oxidation, reforming reactions, and pollution control. The addition of a second metal is an important approach for tailoring the geometric and electronic structures of NPs, thus promoting their catalytic activity and selectivity<sup>14-15</sup>.

Preparation of bimetallic nanoparticles from metal salts can be divided into two groups; co-reduction and successive reduction of two metal salts. Co-reduction is the simplest preparative method of bimetallic nanoparticles, that is, the same as monometallic nanoparticles. The only difference is the number of metal precursors. The successive reduction is usually carried out to prepare core-shell structured bimetallic nanoparticles. Other methods, such as preparation from double complexes and electrolysis of bulk metal.<sup>16-18</sup>

The basic methods of preparing bimetallic materials can be broadly classified into the following six categories, viz (i) chemical reduction, (ii) thermal decomposition of appropriate precursors, (iii) electrochemical syntheses, (iv) radiolysis and (v) sonochemical synthesis. Among these methods, the chemical reduction method can be further sub-classified into (a) co-reduction, (b) reduction of bimetallic metal complexes and (c) successive reduction. Thetwo different nanoparticles cause an increase in specific performance such as electronic and catalytic effects and create synergetic effects<sup>19</sup>. Nickel nanoparticles (Ni-NPs) have many important applications as catalysts, conducting and magnetic materials<sup>20</sup>, and an electrode layer in multilayer ceramic capacitors<sup>21-22</sup> and potential applications in a variety of fields, including electronics<sup>23</sup>, and biomedicine<sup>24</sup>. Ni-NPs can be synthesised using many methods, including photolytic reduction Tseng and Chen<sup>25</sup> (2006), radiolytic reduction, solvent extraction reduction, and microwave irradiation, the size and shape of the products were characterized by various followed by kinetic studies<sup>26-29</sup>.

The objective of this work is to produce bimetallic Ni-Mn nanoparticles supported on graphene oxide layers to combine their properties and observe their catalytic activity to use has a catalyst at room temperature with the help of reducing agent like sodium borohydride. In the light of above advantages and considering the significance of the bimetal nanocatalyst the present study mainly focused with the following objectives, it is proposed to synthesis of two different types of Graphene oxide-supported mono and bimetallic nanoparticles catalysts. Further the individual size characterization of each catalyst was determined using different instrumental techniques Viz.; UV-Visible, SEM, XRD etc.

## 2. MATERIALS AND METHODS:

Graphite powder (SRL), Nickel sulphate, manganese sulphate, sulphuric acid, hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), potassium dichromate (SRL), sodium boro hydride (SRL), sodium hydroxide (Alfa aesar), ethanol (SRL), Double distilled water were all as analytical grade of 99% purity and used as received. The Characterization of the synthesized bimetallic Nanoparticles catalyst characterized by XRD, SEM and UV spectra.

Graphene oxide consists of graphene sheets decorated mostly with epoxide and hydroxyl groups. Rapid heating of GO results in expansion and delamination, due to evaporation of the intercalated water and evolution of gases from pyrolysis of the oxygen containing functional groups. The structure and properties of GO depend on the synthesis method and degree of oxidation. The most common chemical method of synthesis is hummer's method. This method of synthesis varied by varying the oxidizing agents used to exfoliate graphite flakes. Here, we represent two

methods of synthesis technique one is by common method and other by modified method of synthesis namely, Hummer's method. Graphite flakes (5g) and NaNO<sub>3</sub> (2.6g) were mixed in 200ml of H<sub>2</sub>SO<sub>4</sub> in a 1000ml beaker. They stirred for one hour and 20g of KMnO<sub>4</sub> added gradually and stirred for 5days continuously. Finally, the solution colour changed to brown after 5days. 20ml of 30% H<sub>2</sub>O<sub>2</sub> added to terminate the reaction, which is detected by appearance of yellow colour. A 200ml of water added to achieve pure yellowish colour liquid. Then allowed to settle down and decanted thrice followed by several washes; and centrifuged and filtered with 10% HCl and washed with distilled water several times. After filtration, dry under vacuum at room

Graphiteoxide prepared by the using Hummer method and exfoliated into graphene oxide by sonication in water. These Go-supported mono and bimetallic nanoparticles catalyst prepared by chemical reduction method. The first catalyst viz.; Ni-Go bimetallic NPs was prepared by taking in a 100ml round bottom flask, typically, 0.06mm of Nickel sulphate to which Manganese sulphate added. Sonicated 5mg and 30ml water for 30 minutes, GO was added to the above mixture and the reaction was stirred for a further 2 h at 120°C. Finally, 0.1M sodiumborohydride solution added to the reaction mixture. The resulting solution cooled and centrifuged at 2500rpm. The water and ethanol used for further purification. The collected product was dried at 70°C in a vacuum oven. Using similar procedure, by adopting the same quantity of the reagents and experimental procedure the bimetallic and monometallic nanoparticles catalysts viz.; Ni/Mn, Go, Ni and Mn NPs were also prepared. The metal precursors for Ni/Mn, Go, Ni and Mn NPs were MnSO<sub>4</sub>, and NiSO<sub>4</sub>.7H<sub>2</sub>O solution. The reduction of Mn<sup>+</sup>/Ni<sup>2+</sup>, Go, Mn<sup>+</sup>/Ni<sup>2+</sup> to Ni/Mn, Go, Ni and MnNPs was noticed through the change of colour. The resulting two different bimetallic nanoparticles catalysts characterized with UV, SEM and XRD analysis.

The comparative catalytic efficiency of mono and bimetallic nanoparticles catalyst examined by conducting Rhodamine B reduction as a model reaction keeping under identical pseudo-first-order experimental condition. The reaction carried out individually in a standard quartz cuvette (1cm path length) to which 2.5 ml water, 0.25ml Rhodamine B, 0.25ml NaBH<sub>4</sub> (100 mm) and 5mg of respective catalyst were added. After mixing, the respective solution the corresponding cuvette placed in a UV-visible spectrometer at 27°C. The occurrence of the reaction recorded in the range from 200 to 700nm. For example, the UV-visible spectrum recorded for Go-Ni/MnNPs catalyst, the characteristic peaks for the reduction of Rhodamine B observed at 544 nm, which in turn followed gradually decreased through UV-visible spectrometer. The decreasing trend of characteristic peak was recorded at regular time intervals (5min) and similar procedure was used for the calculation of pseudo-first-order rate constant. The pseudo first rate constant was calculated using the following formula.

$$K_{obs} = \ln[A_{\infty} - A_0] / (A_{\infty} - A_t) / t$$

Where  $A_{\infty}$  -absorbance at infinity time,  $A_0$ -initial absorbance,  $A_t$ -absorbance at different time t. The observed  $K_{obs}$  value revealed that the Go-Ni/Mn NPs catalyst found to be superior catalysts compared to other bimetallic and monometallic nanoparticles.

### 3. RESULTS AND DISCUSSION:

The prepared Ni/GO nanocomposites were characterized in detail by means of various spectral and microscopic techniques. A good nanocatalyst should have properties such as strong metal-support interaction, good conductivity, high surface area, good surface morphology and smaller size of nanoparticles with narrow particles size distribution<sup>30-31</sup>. Graphene has emerged as a promising support material for fuel cell electrodes due to its excellent electrical conductivity and unique physical and chemical properties. It is well known that the research on catalysis is proved to be a vital subject and this effort in turn can strengthen the economy of the industries. In the last two decades the research interest on development of homogeneous nanoparticles catalyst has increased tremendously owing to its and unaccountable advantages. To synthesize homogeneous mono/bimetallic nanoparticles catalysts, we need an appropriate template to stabilize/encapsulate the metal nanoparticles.; The present study deals with synthesis of two different methods of graphene oxide supported mono and bimetallic nanoparticles catalysts viz.; Ni/Go, Ni/Mn, Go, Mn and Ni. Graphene oxide is a common stabilizing agent, while MnSO<sub>4</sub> and NiSO<sub>4</sub>.7H<sub>2</sub>O act as a metal precursor for MnNPs, GoNPs, and NiNPs metal nanoparticles through simplified methods. The mono and bimetallic nanoparticles catalysts obtained by different methods were characterized using UV, SEM, and XRD analyses. Previous reportson the synthesis of various mono and bimetallic nanoparticles catalyst obtained through oxidation or reduction of organic substrates. To synthesize homogeneous mono/bimetallic nanoparticles catalysts, we need an appropriate template to stabilize/encapsulate the metal nanoparticles. Further in the preparation of

homogeneous metal nanoparticles as a catalyst, identification of template/stabilizing agent proved to be a bigger task.

Nanoscale materials have attracted significant scientific and industrial interests. Considerable effort has been devoted to nanocomposite studies, that is, alloy and core-shell nanoparticles, because of their valuable properties which make them useful for composition-dependent optical application and catalysis. Bimetallic nanoparticles retain a greater degree of catalytic activity than the monometallic ones. The simple approaches for the fabrication of GO-supported mono as well as bimetallic NPs were synthesized by co-reduction method using NaBH<sub>4</sub> in aqueous medium. The catalyst of GO supported bimetallic NPs were synthesized in three steps. In the first step, the graphene was converted in to graphene oxide by using hummer's method. The synthesis of stable nanoparticles was very difficult without any stabilizing agent. Before addition of sodium boro-hydride the metal ions, the mixture was stirred at least 2h. In step two, the colloidal metal ions solution was injected into the GO solution under stirring condition. Finally, the sodium borohydride solution was added dropwise into the above mixture. The reaction was further continued for 30 minutes in the third step. The obtained products viz.; mono and bimetallic NPs were characterized by UV, SEM, and XRD analysis. The catalytic efficiency of the newly developed NPs was studied for the reduction of organic dyes as a model reaction. The reduction reaction was monitored by using UV-visible spectroscopy and the reusability of the superior catalyst was examined up to 3 cycles for the same reduction reaction.

The electronic properties of Graphene-oxide/Ni-Mn were analyzed by UV-Visible absorption spectral technique. Since, the oscillation energy of surface electrons (plasmas) of GoNps is in resonance with the visible light around (235-245) nm, the GoNPs absorb the visible light around (235-245) nm. The GoNPs can be identified by recording the UV-Visible absorption spectrum. The UV-Visible absorption spectrum of Go/Ni-Mn exhibit two signals at (850-870) nm and (300-400) nm. The absorption signal at (850-870nm) is corresponding to the  $\pi - \pi^*$  transition, while a signal at (300-400) nm is corresponding to the  $n - \pi^*$  transition. These electronic properties are the characteristics of Go/Ni-Mn. The UV-Visible absorption spectra of Go/Ni-Mn exhibit the SPR signal of GoNPs at 220 nm and it confirm GoNPs formation. In addition to the SPR peak at (235-245) nm, the GoNPs with Ni/Mn nanoconjugates exhibit two strong absorption signals at (300-400) nm and (850-870) nm. It confirms the conjugation of GoNPs to beta carotene molecule.

**Fig. 1 UV of Graphene oxide supported with bimetallic**

**Fig .2 represents the SEM image of Ni-Mn/graphene compounds (Ni-Mn)**

The surface morphology of three monometallic nanoparticles catalysts was performed by SEM analysis, and the observed results are compared with corresponding polymer control and SEM image was smooth in surface without any heterogeneity. In contrast the Ni and Mn NPs obtained from Fig 2 reveals that irrespective of the images, there is an evenly distributed white dot appeared on the surface of mono and bimetallic NPs. This must be a contribution of formation of evenly distributed NPs on the surface of the spherical shape. Based on the results, monometal nanoparticles are well supported by GO

The GO supported bimetallic Ni-Mn NPs were obtained after centrifugation and the dried samples were analyzed by powder XRD to determine their crystalline nature and particles size with  $2\theta$  values showed the characteristic peaks for crystalline mono Mn NPs and bimetallic Ni-Go as well as Ni-Mn NPs. The inset of Fig.3 shows that the peak corresponding to LCO NPs was slightly shifted to a lower angle, which revealed that the La was loaded on the surface of CuO material. The crystalline sizes of the LCO and CuO NPs were determined using the Debye-Scherrer equation, where D is the crystal size of the catalyst, K is a dimensionless constant,  $\lambda$  is the wavelength of the X-rays, b is the full width at half-maximum (FWHM) of the diffraction peak and q is the diffraction angle.

**Fig.3 XRD image of Ni-Mn NPs**

#### **4. Kinetic study for the reduction of Rhodamine-B using Graphene Oxide supported Ni-MnNPs catalyst:**

To realize the effect of NaBH<sub>4</sub>, substrate and catalyst on the reduction of organic dyes, the superior catalyst viz.; Ni-Mn NPs was employed and studied under pseudo-first-order reaction condition. As usual, the kinetics of the dye's

reduction reaction was followed by measuring the absorbance of the product with decreasing trend 544 nm against the time through UV-visible spectrophotometer. From the observed rate constants, it is understood that the  $\text{NaBH}_4$ , substrate and catalyst have been largely influenced the  $k_{obs}$  value in the reduction of organic dyes substrate.

#### 4.1 Effect of substrate:

The substrate concentration varied from 0.8, 1.0, 1.2 and 1.4 mm is mention as A, B, C, D at constant temperature  $27^\circ\text{C}$  and the experiments were performed by maintaining the other parameters as constant. The rate constants are calculated from the plot  $2+\log(A-A_t)$  vs time and the calculated values are given in Fig.6. The observed rate constants are decreased invariably on increasing the dyes are given in Table1 and as shown in the Fig.7. The decreasing trend of rate constant is due to lesser availability of  $\text{BH}_4^-$  in aqueous phase and thus minimizing the formation of product.

Fig.4  $K_{obs}$  vs [Rh-B]

Table 1. Effect of [substrate]

S. No	[Rh-B] mm	[ $\text{NaBH}_4$ ]mm	[Catalyst]mg	$K_{obs}\times 10^{-3}\text{s}^{-1}$
1.	0.8	100	5	0.407
2.	1.0	100	5	0.380
3.	1.2	100	5	0.364
4.	1.4	100	5	0.342

#### 4.2 Effect of [ $\text{NaBH}_4$ ]:

The effect of concentration of  $\text{NaBH}_4$  for reduction of rhodamine b was studied in the range from 60, 80, 100 and 110mm is represent as A, B, C, D keeping the other parameters at constant. From the plot of  $2+\log(A-A_t)$  vs time, the pseudo-first-order rate constants were evaluated and the corresponding plots are shown in Fig. 8. The observed rate constants increased with the increase in the concentration of  $\text{NaBH}_4$  and presented in Table 2 and the plots are depicted in Fig. 9. On increasing [ $\text{NaBH}_4$ ], the adsorption of  $\text{BH}_4^-$  ions onto the nanoparticles surfaces also increased parallelly; as a result, the relay of electrons from  $\text{BH}_4^-$  (donor) to the Rh-b (receptor) increased. It is worth to state that under the fixed concentration of  $\text{NaBH}_4$ , the reduced product viz.; organic dyes remain non-oxidized, since the liberated hydrogen eliminates the air hence prevents the oxidation. In addition, the evolved hydrogen bubbles from the catalyst surface helps in mixing of the solution and thus offering the favorable conditions to increase the reaction rates.

Fig.5  $K_{obs}$  vs [ $\text{NaBH}_4$ ]

Table 2. Effect of [ $\text{NaBH}_4$ ]

S. No	[Rh-B] mm	[ $\text{NaBH}_4$ ]mm	[Catalyst] mg	$K_{obs}\times 10^{-3}\text{s}^{-1}$
1.	1.0	60	5	0.37698
2.	1.0	80	5	0.37977
3.	1.0	100	5	0.38248
4.	1.0	110	5	0.40136

#### 4.3 Effect of catalysts:

Similarly, in the case of change in the catalyst concentration, the  $k_{obs}$  found to increase with respect to the concentration of catalyst irrespective of organic dyes used. The effect of GO-Ru-Pd NPs, was performed by varying the concentration of the catalyst from 3, 5, 7 and 8 mg is represent as A, B, C, D which is shown in Fig, 10, by keeping the other parameters constant. The plot derived from observed rate constant against the amount of catalyst was shown in Table, 3. From this plot, it has been understood that the amount of catalyst and the rate constant are parallel to each other are given in Fig, 11. It has been obviously known fact, that at higher concentration of the catalyst, increases active sites of nanoparticles which enhances the rate constant.

Fig.6  $K_{obs}$  vs [catalyst]

Table 3. Effect of catalysts

S. No	[Rh-B] mm	[ $\text{NaBH}_4$ ] mm	[Catalyst] mg	$K_{obs}\times 10^{-3}\text{s}^{-1}$
1.	1.0	100	3	0.34275
2.	1.0	100	5	0.37812
3.	1.0	100	7	0.39478
4.	1.0	100	8	0.41153

## 5. CONCLUSION:

Different types of graphene oxide supported two mono Ni, Mn and bimetallic Ni/Mn nanoparticles catalyst. Graphene oxide supported mono and bimetallic nanoparticles were synthesized by co-reduction method using NaBH<sub>4</sub> in aqueous medium. The obtained both mono and bimetallic nanoparticles catalysts were shape and size characterized by using various techniques such as, scanning electron microscopy (SEM), X-ray diffraction spectroscopy (XRD). From the observed results it reveals that the newly developed graphene oxide supported bimetallic nanoparticles catalysts can be more efficient to reductive, oxidative and of environmentally important organic pollutant additionally it is also very good biologically active compound.

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