



**BIODIRECTED SYNTHESIS OF Ag-Ni ALLOY NANOPARTICLES FROM MARINE ALGAE (CLADOPHORA) AND IT'S APPLICATION IN PHOTOCATALYTIC DEGRADATION OF CRYSTAL VIOLET**

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**ABSTRACT**

In bio organisms algae is one of the important source of reducing and capping agents for the synthesis of bimetallic nanoparticles (NPs) due to simple procedure and inexpensive cost required. This paper reports the biosynthesis of Ag-Ni alloy bimetallic NPs by using marine algae (*Cladophora*) as reducing and capping agent. The morphology, size and composition of Ag-Ni NPs were confirmed by scanning electron microscope (SEM), energy dispersive x-ray analysis (EDX) and transmission electron microscopy (TEM) analysis. The biosynthesized Ag-Ni NPs was used for the photocatalytic degradation of environmental pollutant crystal violet under solar light. The optimum conditions for the degradation were measured with UV-Visible spectral analysis. Due to the smaller size of Ag-Ni NPs exhibit great photocatalytic performance.

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**INTRODUCTION**

In the last few years the field of nanotechnology has received an immense attention due to its vast applicability from the kitchen to space. 'Nanotechnology' is the application of science to control the matter at molecular level [1]. It involves the synthesis and application of materials having one of the dimensions in the range of 1-100nm, thus act as a bridge between bulk materials and atomic or molecular structures [2]. Recently, bimetallic nanoparticles have received considerable attention because of their importance for magnetic, optical and catalytic applications in variety of fields [3- 5]. Bimetallic nanoparticles have characteristic mixing patterns and geometrical architecture which enhances their functionality [6- 8]. Among wide range of bimetallic systems Ag-Ni nano compositions have gained significant advancement in the field of magnetic, optical, biological and anti-oxidation properties [9- 11]. To synthesize bimetallic nanoparticles, a physical method, a chemical method or biological method has been implemented. Several advantages of biological methods over physical and chemical are that the process are environmental friendly, less time taking, almost negligible industrial waste, no use of toxic chemicals. As both the synthesis and applications of nanoparticles are important, many researchers from both academic and industry are focusing towards biological systems such as plants, marine algae, fungi and bacteria for the production of nanoparticles [12- 15]. Seaweed is one of the most extensively used functional foods and

medicinal herbs, the use of which dates back at least 5,000 years to ancient china [16]. Marine algae refer to a wide variety of different species with different medicinal behavior that can be divided into two groups, namely, micro algae and macro-algae [17]. Marine algae contain more than 60 trace elements with higher concentration than the terrestrial plants and also contain proteins, iodine, vitamins, polysaccharides [18] and they produce great variety of secondary metabolites. These secondary metabolites act as reducing as well as stabilizing agent for the synthesis of metallic nanoparticles [19, 20].

The organic dye effluents from textile, paper, plastic, medicine and photographic industries are becoming a major threat to the biodiversity because of their toxicity, unacceptable color, high chemical oxygen demand content, and resistance to chemical, photochemical, and biological degradation [21]. A necessary criterion in the use of these dyes is that they must be highly accumulated in water and stable in light during washing. The accumulation of these dyes in the water bodies causes eutrophication, reduces the reoxygenation capacity and makes severe damage to the aquatic organisms by hindering the infiltration of sunlight [22,23].

Crystal violet (CV) is a triarylmethane synthetic dye which is widely used in industries such as textile/ dyeing, ball-point pen, paper, leather, additives, foodstuffs, cosmetics [24- 27]. Investigations indicate that crystal violet has carcinogenic, toxic and mutagenic for water organisms and mammalian cells [28- 30]. Several methods were being proposed to remove dyes from the textiles viz. physical, chemical and biological methods. Therefore there is a need to develop a novel

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treatment method that is highly effective in eliminating crystal violet from the waste water.

Nano materials play an important role in the degradation of organic dyes [31, 32]. Biologically synthesized Ag, Ni, NiO and Fe-Ni nanoparticles play a role in the degradation of crystal violet [33- 37]. Here, we report the novel synthesis of Ag-Ni nanoparticles by using marine algae *Cladophora* and its use in the photo degradation of crystal violet.

## MATERIALS AND METHODS

The biosynthesis of Ag-Ni alloy NPs from Marine algae (*Cladophora*) was carried out as described below.

### Materials

*Cladophora* algal extract, Whatman number 1 filter paper, distilled deionized water, silver nitrate  $\text{Ag}(\text{NO}_3)_2$  and nickel nitrate hexahydrate  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  commercially obtained from Sigma Aldrich Company were used.

### Collection of Algae and preparation of algal extract

The marine green Algae, *Cladophora* was collected from the Visakhapatnam coastal area, Andhra Pradesh, India, in the month of April. The marine green seaweed was soaked in water for 1hour to remove the sand and mud and then thoroughly washed with fresh water and distilled water to remove the salt minerals and metallic compounds on the external part of the seaweed. Clean seaweed was dried at a shady place for ten days i.e. until the weight of the dried seaweed remains constant. The dried seaweed was ground into fine powder. 10 g of algal powder was mixed with 200 mL of distilled water in the 500mL beaker and boiled, extract was filtered through Whatman Filter Paper No.1, collected the supernatant and stored at 4° C for nanoparticle synthesis.

### Biosynthesis of Ag-Ni nanoparticles from algal extract

Biosynthesis of bimetallic Ag/Ni nanoparticles was carried out as follows. 30mL of *Cladophora* algal extract was added drop wise to 170mL of a 1:1 4mM solution of  $\text{AgNO}_3$  and  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  in Erlenmeyer flask under continuous stirring for 30min. The set up was kept in dark under room temperature for night for the completion of reduction of  $\text{Ag}^+$ - $\text{Ni}^{2+}$  to Ag-Ni. The progress of the reaction was monitored by recording the spectra from 200-800nm SHIMADZU UV-2450 double beam UV-Vis spectrophotometer operated at 1nm resolution. The bimetallic Ag/Ni nanoparticles were obtained by repeated centrifugation of the resulted colloidal solution at 8000rpm for 15min. The obtained pellets were washed thoroughly and re-dispersed in deionized water dried in an oven at 80° C.

### Characterization

The formed Ag/Ni bimetallic nanoparticles structure and composition were examined by scanning electron microscopy (SEM, Hitachi S-3700N), energy dispersive X-ray spectroscopy (EDS, sigma).

## RESULTS AND DISCUSSIONS

### UV-Visible spectroscopic studies of Ag/Ni bimetallic nanoparticles

Figure 1 depicts UV-Visible absorption spectra in the range of 200-800 nm for biologically synthesized Ag/Ni bimetallic nanoparticles. The characteristic band is appeared at 398 nm

represents surface plasmon resonance (SPR) characterized by Ag/Ni NPs and was not obtained by bulk materials [38, 39].

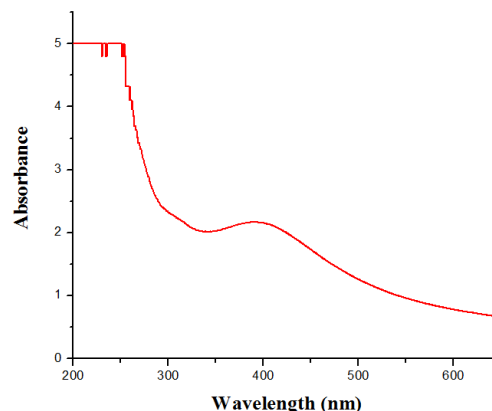


Figure 1 UV-Visible spectra of Ag/Ni NPs.

### Scanning electron microscope studies

The morphology of biosynthesized bimetallic Ag/Ni nanoparticles were studied by using a scanning electron microscope (SEM, Hitachi S-3700N). The images obtained at different magnifications were shown in Figure 2. The SEM images clearly indicated that the size of Ag/Ni bimetallic nanoparticles are in spherical and in the range of 50-110 nm morphology.

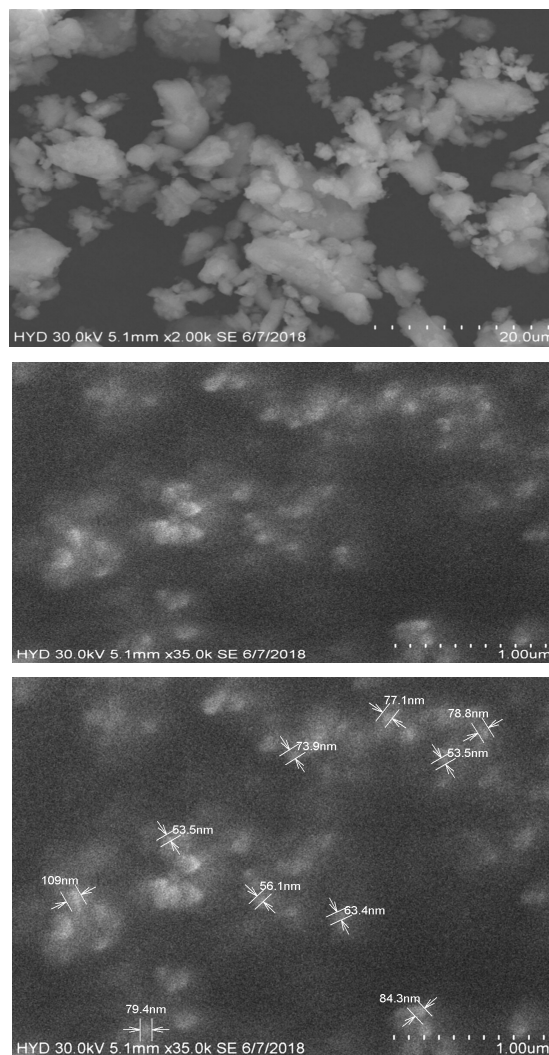


Figure 2 SEM images of Ag/Ni bimetallic NPs.

### Energy dispersive X-ray analysis

Energy dispersive X-ray was used to study basic component of biosynthesized bimetallic Ag/Ni nano sample and their relative weights. The EDX spectra recorded was shown in figure 3. It was observed that carbon from used algal extract is the basic that represent major fraction of the sample and the other peaks in the figure represent silver and nickel nanoparticles.

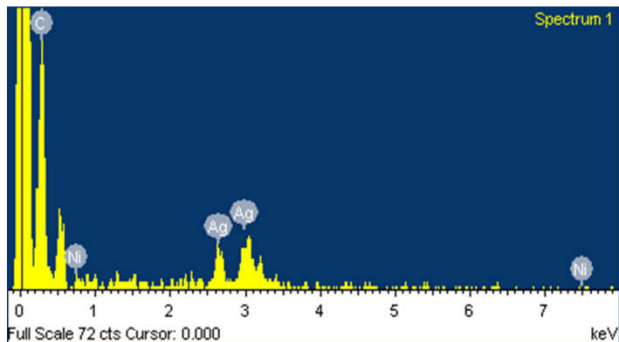


Figure 3 EDX spectra of biosynthesized Ag/Ni alloy NPs.

Table 1 Quantitative results of Ag-Ni NPs.

Element	Weight%	Atomic%
C K	63.06	93.27
Ni L	4.65	1.41
Ag L	32.29	5.32
Totals	100.00	

### Transmission Electron Microscopy studies

The transmission electron microscopy (TEM) images along with selected area electron diffraction (SAED) pattern of biosynthesized Ag-Ni NPs were shown in Figure.4. It was shown that the nanoparticles are crystalline and spherical in shape.

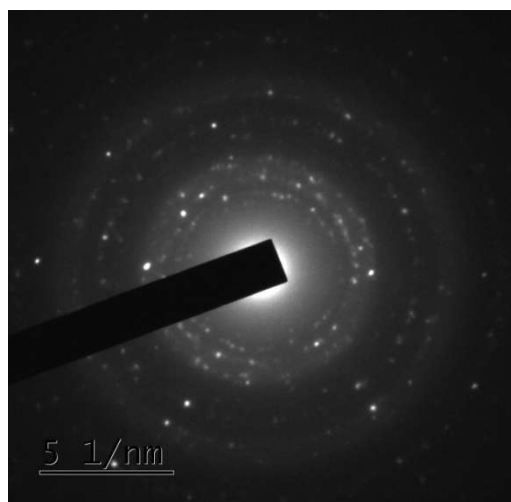
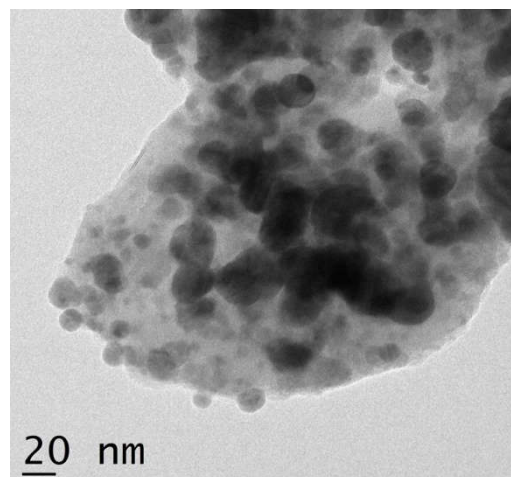
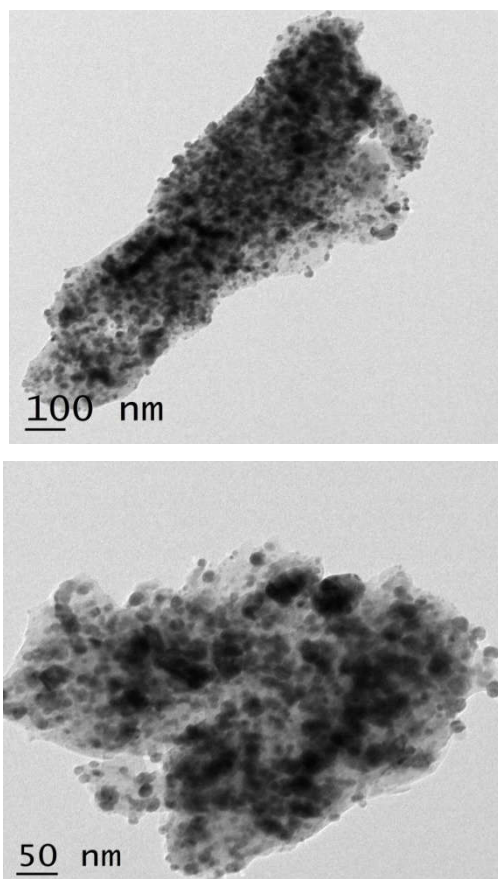


Figure 4 TEM at different magnifications and SAED images

### Photo Catalytic Studies of Ag-Ni Bimetallic Alloy NPs on the Degradation of Crystal Violet

The photocatalytic activity of biosynthesized Ag-Ni alloy NPs was studied by photo degradation of crystal violet. The aqueous solution of crystal violet was found to be stable even after 90min in the absence of photocatalyst. There was a decrease in the concentration of dye solution in the presence of Ag-Ni alloy NPs. Initially, the 10 ppm of dye solution was prepared by addition of 0.001g of crystal violet in 1L of deionized water. To 100mL of 10ppm concentrated dye 0.05mg of biosynthesized Ag-Ni alloy nanocatalyst was added. The maximum absorbance of crystal violet was found at 580 nm by UV-Vis spectrophotometer. The initial intensity of crystal violet solution was measured before the addition of nanocatalyst. The pH of the solution was balanced by the addition of 0.1N NaOH or 0.1N HCl. The suspension was kept under magnetic stirrer continuously in dark for 45min to establish the adsorption-desorption equilibrium. For this experiment sun light acts as a major source. The setup was kept under sunlight in between 11am to 3pm in May. At frequent intervals (every 15min) of given irradiation time, 5mL of suspension was collected and centrifuged to remove the particles. The resultant solution was monitored in the wavelength range 200-800nm in a UV-Visible spectrophotometer. Distilled water is used as a reference. The absorbance of crystal violet in solution as a function of time is shown in Figure 6 shows the decrease in the intensity of peak

at 580 nm due to the decrease in the concentration of crystal violet in the solution on solar irradiation.

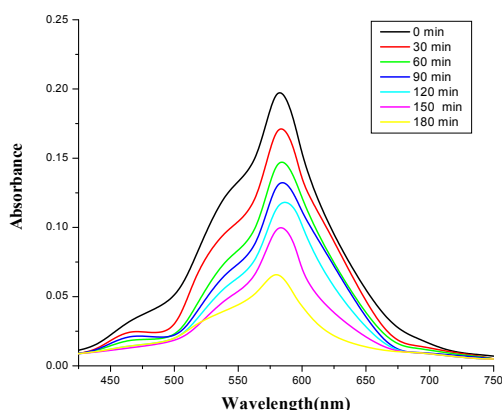


Figure 6 The photodegradation of dye as a function of time.

The percentage of photodegradation was calculated by using equation (1):

% degradation =

$$\left( \frac{A_0 - A}{A_0} \right) \times 100 \quad (1)$$

Where A is the absorbance at time t and A<sub>0</sub> is the dye initial concentration before degradation

### Effect of pH

One of the important parameter which affects the efficiency of catalyst towards photodegradation is pH of the solution. The effect of pH on the degradation of crystal violet was studied in the pH range of 4-10 at constant initial dye concentration and catalyst dosage. The results were shown in the fig 2. It has been observed that the percent degradation of the dye increases with increase in pH from 4-8. In acidic solutions the surface of the catalyst is positive and the adsorption of cationic dye on the surface is less. The degradation efficiency again decreased from pH=8 to pH=10 may be due to the neutralization of cationic form of crystal violet dye which does not involved in any attraction with the neutral or charged surface of the catalyst [40,41]. The effect of pH on percentage of photodegradation of dye is shown in Figure 7.

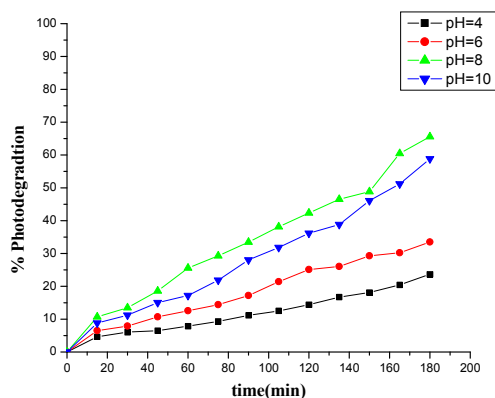


Figure 7 Effect of pH percent on percentage of degradation.

### Effect of catalyst loading

Catalyst loading is one of the important factors that affect the photocatalytic degradation rate. The effect of catalyst loading was studied by varying the catalyst concentration from 0.025g-0.1g at pH=8 with dye concentration 10ppm. The results indicated that the degradation rate increases with increase in catalyst loading from 0.025 to 0.05g. This is due to an increase in number of Ag/Ni nanoparticles, which increases the absorption of photons and adsorption of dye molecules. Further increase in catalyst loading from 0.05 - 0.1, decreases the degradation rate. This may be due to the screening effect of catalyst particles. At high concentration of catalyst, the catalyst particles act as screen for the light to travel into the solution. Moreover at high concentrations of catalyst, the part of the catalyst surface becomes unavailable for the photon absorption due to the agglomeration of catalyst particles. The optimum weight of catalyst loading was 0.05g at pH 8 and dye concentration 10ppm [42, 43]. Effect of catalyst loading on percentage of degradation is shown in Figure 8.

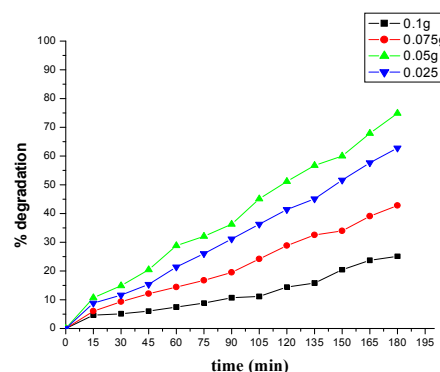


Figure 8 Effect of catalyst loading on percentage of degradation.

### Effect of dye concentration

The effect of dye concentration was studied by varying the initial concentration from 10ppm to 25ppm with catalyst 0.05g and at pH=8. It was observed that initially the % degradation was increased from 10ppm to 15 ppm. This is due to the fact that as the concentration of crystal violet solution increases, more crystal violet molecules are available for the reaction. Further the % of photodegradation was decreased with the increase in concentration of crystal violet solution. At high dye concentrations more dye molecules absorb on the catalyst surface, decreases the number of active sites for the reaction [44]. The effect of initial dye concentration on percent degradation is shown in figure 8.

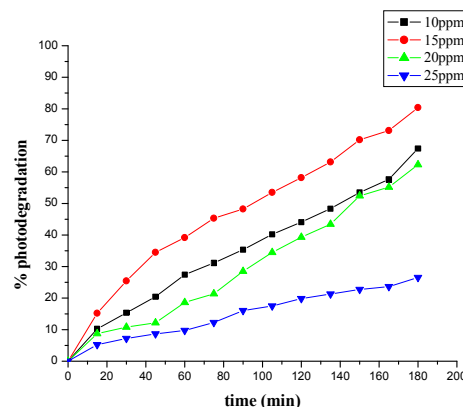


Figure 9 The effect of initial dye concentration on percent degradation

## CONCLUSIONS

Biosynthesis of Ag-Ni alloy NPs with the size below 100nm was successfully obtained by using marine algae (*Cladophora*). Ag-Ni alloy nanoparticles formed are crystalline and spherical in shape. The degradation of environmental pollutant crystal violet can be successfully achieved up to 80% by using biosynthesized Ag-Ni nanoparticles. The % photodegradation was increased from pH 4 to 8 and decreased from pH=8 to 10. The degradation efficiency of nanocatalyst was increased from 0.025g to 0.05g and again decreased from 0.05g to 0.1g. The percent degradation was initially increased with dye concentration from 10ppm to 15ppm, further the percent degradation was decreased with increase in dye concentration from 15ppm to 25ppm. The optimum conditions for the degradation of crystal violet by using Ag-Ni alloy NPs was pH=8, 0.05g of catalyst and 15ppm of dye concentration.

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