Comparative Structural Studies of the Silver and Nickel Nano Composites Synthesized by Chemical Methods

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Abstract

Silver and nickel nanoparticles were synthesized from urea and formaldehyde by using chemical method, followed by calcination at 8000C. The chemical composition and crystallographic structure of silver and nickel nanoparticles were confirmed by XRD. Surface imaging studies like AFM was carried out for understanding surface morphology and particles size distribution. The spherical and porous-like morphology of silver and nickel nanoparticles were confirmed by SEM and TEM. White spots are observed because of the the presence of silver and nickel metal ions in spherical forms. The particles size of silver and Nickel metal ions obtained through XRD are found to be 41.43 nm and 13.71 nm respectively.

Introduction

Polymeric materials are widely used in industry due to their ease of production, lightweight and ductile nature. A very effective approach to improve mechanical properties of polymer is to add fibers, whiskers, platelets or particles as reinforcements to the polymer matrix. The polymer nanoparticles show enhanced properties after the incorporation of low amount of Nano fillers such as carbon black (CB), carbon nanotubes(CNTs), graphene and Nano clay (Huang, 2002, Moniruzzaman et al. 2006, Kim et al. 2010,Fischer, 2003). Polymers have been filled with several inorganic compounds, either synthetic or natural, in order to increase heat and impact resistance, flame retardancy and mechanical strength, and to decrease electrical conductivity and gas permeability with respect to oxygen and water vapor (Hajji et al. 1999). Such composites are widely used in many areas like in electronics, catalysis, transportations and construction, because of their novel properties. Compared to conventional composite nanoparticles, polymer nanocomposites have ultrafine nanometer size phase dimensions and offer unique combination of properties due to the size (Sanchez et al. 2000, Sanchez et al. 1999, Pomogilo, 2000, Novak, 1993, Lichtenha et al. 2001, Sanchez et al. 1994, Ells et al. 1999, Kwiatko et al. 2000, Schubert et al. 1995, Mori Kawa et al. 1992, Giannelis et al. 1999, Jordan et al. 2005). The properties of polymers can be improved by doping with metal nanopartilces (Akita et al. 1999, 1999, 1999, Chang et al. 2002, Zavyalov et al. 2002). Another important aspect of nanoscale reinforcement is that it has exceptional potential to generate new phenomena giving rise to special properties in these materials. Urea- Formaldehyde (UF) resin is widely used as thermosetting materials due to low cost and good thermal and mechanical properties including chemical resistance. This resin, which is the condensation product of urea and formaldehyde, are used with reinforcing fillers or fibers to produce composite materials for use in a wide range of commodity, engineering and other special applications. Several preparative methods of composite particles of polymer and magnetite particles he been proposed (Ma et al. 2005) like suspension polymerization (Horak et al. 2010), dispersion polymerization (Pimpha et al. 2015), emulsion polymerization (Wang et al. 2005) soap-free emulsion polymerization (Mori et al. 2007) mini emulsion polymerization (Liu et al. 2003) and micro emulsion polymerization (Chaudhary et al. 2017). All these methods of nanoparticle formation require high temperature and expensive instruments. One of the simplest and cost effective techniques to prepare the silver and nickel nanoparticles is by thermal decomposition. This technique has various advantages over other method. These include a reaction that can be easily controlled requiring short reaction time for the preparation of different type of nanoparticles (Traversa et al. 1998, Farhadiet al. 2010).

Amongst the metal nanoparticles, Ag NP is becoming an increasingly important material in many technologies. Ag NPs exhibit the highest efficiency of Plasmon excitation and is the only material whose Plasmon resonance can be tuned to any wavelength in the visible spectrum. Ni NPs have also been getting importance because of its application as catalysts and conducting and magnetic materials (Kulkarni, 2015). The objective of this study has been to synthesize silver and nickel nanoparticle by using urea and formaldehyde resin as precursors, followed by thermal decomposition. The synthesized nanoparticles have been characterized using Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and Atomic Force Microscopy (AFM).

Material And Methods

Silver nitrate ($AgNO_3$), Nickel Chloride Hexahydrate ($NiCl_2.6H_2O$), Formaldehyde (HCHO) and Urea (NH_2CONH_2) were purchased from Central Drug House Pvt. Ltd. and Sodium Hydroxide (NaOH), hydrochloride Acid (HCl) were purchased from Fisher Scientific. All solvent was of analytical reagent grade and were used without further purification for the preparation of metal solutions.

Silver and Nickel nanoparticle were synthesized by the following steps:

Step1: Formation of polymer metal complex

Step2: Formation of silver/ nickel nanoparticle

Step 1: Formation of Polymer metal complex

1.38 mole of urea, 1.13 moles of formaldehyde and 15 ml glacial acetic acid add in three- necked round bottom flask equipped with reflux condenser and stirrer. The mixture was agitated and was allowed cool off. Water was removed by slowly raising the temperature to 60^0Cand applying a vacuum by means of water pump. This temperature was maintained till the melting of sample which on cooling solidifies into a white colored solid. Then 15 ml of silver and nickel metal solution were added to prepare polymer metal complex respectively. The reaction is exothermic in nature. The dried solid sample was purified by washing with distilled water.

Step2: Formation of silver and nickelnanoparticles:

The polymer metal complex calcinated at 800^0C for 45 minutes in muffle furnace. Black colored silver and nickel nanoparticles were obtained (Chaudhary et al. 2017, 2018).

Purification of silver and nickel nanoparticles:

Metallic ions were removed from the nanoparticle by keeping it in 12N hydrochloric acid solution for 24 hours. The mixture was centrifuged and washed with distilled water till hydrochloric acid was completely removed (Chaudhary et al. 2018). The volatile impurities got separated at time of calcination.

Result And Discussion

Morphological Studies:

SEM images of silver nanoparticles are shown in Fig. 1 ((A and B). The grey coloured bulk regions indicate bulk polymer matrix and white spherical spots are the distribution of silver nanoparticles. The surface is completely rough due to homogeneous distribution of silver particles which are aggregated on polymer surface.

SEM images of nickel nanoparticles are shown in Fig. 2(A andB). Spherical pores are observed on the polymer surface. Interestingly, the morphology of nickel nanoparticle show the carbon black porous like structure and some particles show granular type structure.

Microstructural characterization studies were conducted to determine the size of nanoparticles, homogeneity and size distribution. TEM images of silver nanoparticles as shown in Fig. 3 (A) and (B) respectively show globular as well as rod-like structure on the polymer surface. The black coloured spot on polymer surface indicate metal particles and white bulk part is polymer matrix. Figure 4 (A) and (B) are the TEM image of nickel nanoparticles. This shows bulk granules like morphology. The black colour present in polymer surface are metal particles and white part is polymer matrix. Thus, Ag NPs were recorded to be globular and rod like whereas nickel nano particles are entirely of granular morphology.

X-ray Diffraction (XRD) Analysis:

Table 1
XRD peak list of silver nanoparticles

Pos.[°2Th.]	Height[cts]	FWHMLeft[°2Th.]	d-spacing[Å]	Rel. Int.[%]
32.4336	62.16	0.2362	2.76052	2.55
38.3467	2442.12	0.2362	2.34736	100.00
44.5116	976.67	0.2657	2.03552	39.99
46.4111	29.77	0.3542	1.95654	1.22
64.6085	364.99	0.2657	1.44258	14.95
77.5439	290.57	0.1771	1.23109	11.90

Table 2
XRD peak list of nickel nanoparticles

Pos. [°2Th.]	Height[cts]	FWHMLeft[°2Th.]	d-spacing[Å]	Rel.Int.[%]
43.5115	63.77	0.4723	2.07996	100.00
50.4315	26.39	0.9446	1.80959	41.39

FWHM	FWHM in radian	2Theta	Theta	angle in radian	COS	d (A)		Calculated Size
0.1771	0.003089411	16.49	8.245	0.143829444	0.989674364	453.4865157		
0.2066	0.003604022	32.0382	16.0191	0.2794443	0.961208861	400.246163		
0.4723	0.008239011	39.5195	19.75975	0.344697861	0.941177592	178.8075019	414.3494	41.43 nm
0.1181	0.002060189	45.7766	22.8883	0.399273678	0.921343594	730.4722829		
0.1771	0.003089411	56.848	28.424	0.495840889	0.87956895	510.2544595		
0.3542	0.006178822	66.5049	33.25245	0.580070517	0.836424003	268.2873624		
0.4723	0.008239011	75.5318	37.7659	0.658805144	0.790724253	212.8297106		

Table 3
Calculations of XRD for Silver Nanoparticles

FWHM	FWHM in radian	2Theta	Theta	angle in radian	COS	d (A)		Calculated size
0.4723	0.008239011	43.5115	21.75575	0.379516972	0.928843692	181.1818452	137.0898	13.71 nm
0.9446	0.016478022	50.4315	25.21575	0.43987475	0.904805005	92.99772498		

Table 4
Calculations of XRD for Nickel Nanoparticles

As shown in Tables 3 and 4, the calculated size of the synthesized silver and nickel nanoparticles are 41.43 nm and 13.71 nm respectively which corroborates efficacy of the synthetic method for the preparation of nanoparticles.

Atomic Force Microscopy (AFM) Analysis:

Surface imaging studies were performed using AFM for estimating surface morphology and particles size distribution. Spherical shaped metal ions were identified at the surface of silver nanoparticles. The white spots as shown in Fig. 7 (A and B) and 8 (A and B) represent the presence of silver and nickel metal ions respectively in spherical forms.The maximum peak height obtained is of 37.7 nm and 92.1 nm for silver and nickel nanoparticles respectively.

The present study supports the findings of (Kotakadi et al. 2013) who have also made the similar observations that AgNPs have a spherical shape and the diameters of AgNPs varied between 27 and 50 nm. Similarly, (Muzamil et al.2014) have also recorded that particles are nearly spherical identical in shape, dispersive & crystalline in structure.

Conclusion

Silver and nickel nanocomposites were synthesized by chemical precipitation method followed by thermal decomposition which is greener and environmentally suitable, cheap and time-saving as compared to conventional methods.The morphology studies by SEM show that the silver nanoparticles are spherical and nickel nanoparticles have pore-like shapes like that of carbon-black. The particle sizes were determined by XRD and compares well with previous studies and surface to volume area results of AFM studies also indicate the exact structure and probable properties of the synthesized nanoparticles.Our results are good indicative of the efficacy of the chemical precipitation method in the synthesis of the nanoparticles.In future, these Silver nanoparticles can be further analyzed for its heat transfer properties and application in nanofluids and conductive gels. It is also proposed to investigate the catalytic activity of nickel nanoparticles in future.

Abbreviations

AgNPs: Silver Nanoparticles; Ni NPs: Nickel nanoparticles; SEM: Scanning Electron Microscopy; TEM: Transmission Electron Microscopy; AFM: Atomic Force Microscopy; NPs: Nanoparticles; XRD: X-Ray Diffraction; 3D: Three Dimensional; 2D: Two Dimensional; CB: Carbon Black; CNT: Carbon Nano Tube; UF: Urea-Formaldehyde;

Declarations

Availability of data and materials

Not applicable. The corresponding author may be contacted for data.

Competing interest

The authors declare no conflict of interest.

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Author contributions

Giriraj Tailor: Conceived and designed the experiments; Analyzed and interpreted the data; Drafted the paper.

Jyoti Chaudhary: Contributed reagents, materials, analysis tools for data.

Bhupendra Kr. Sarma: Provided lab facilities for carrying out the experiments and checked the draft of manuscript.

Deepshikha Verma: Performed the experiments.

Conflicts of Interest:

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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Figures

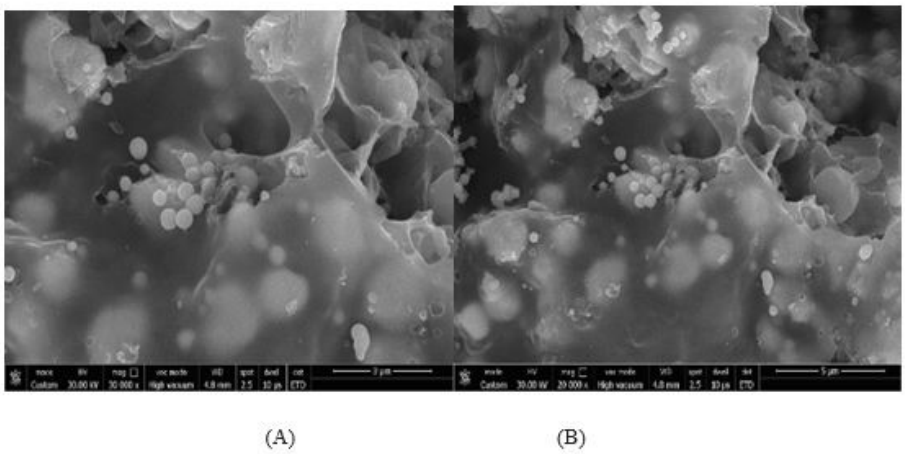

Figure 1

SEM images of silver nanoparticles (A) 30000× (B) 20000× Magnification

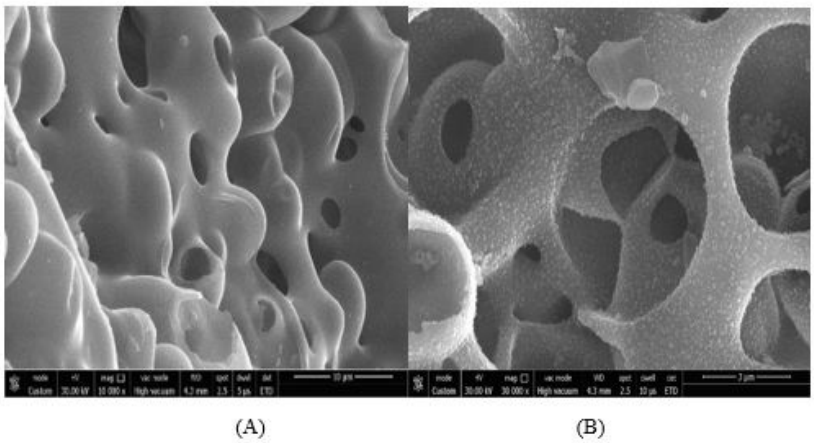

Figure 2

SEM images of nickel nanoparticles (A) 10000× (B) 30000× Magnification

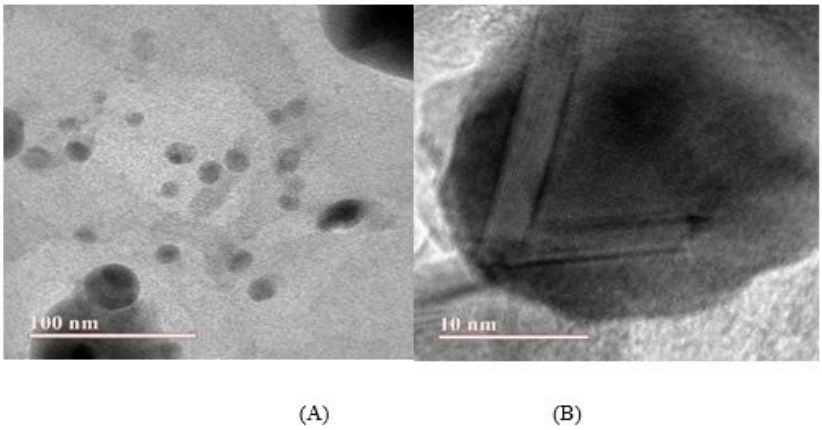

Figure 3

TEM images of Silvernanoparticlesat (A) 100nm and (B) 10 nm

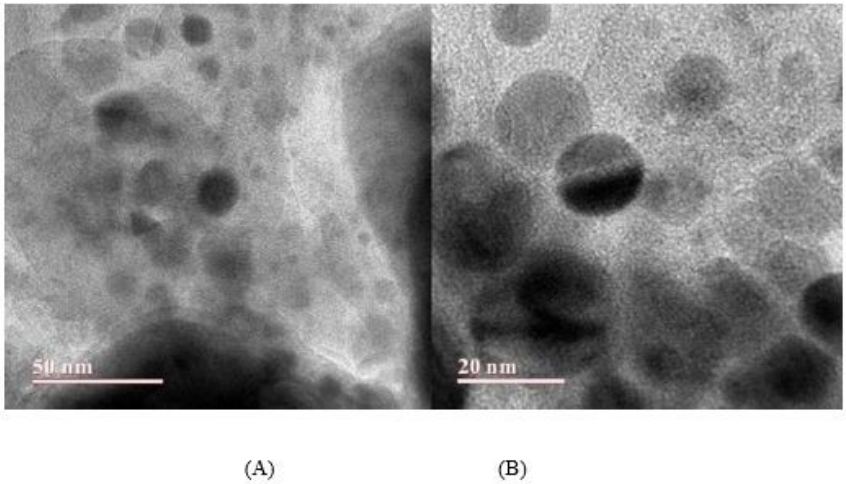

Figure 4

TEM images of Nickel nanoparticles at (A) 50nm and (B) 20 nm

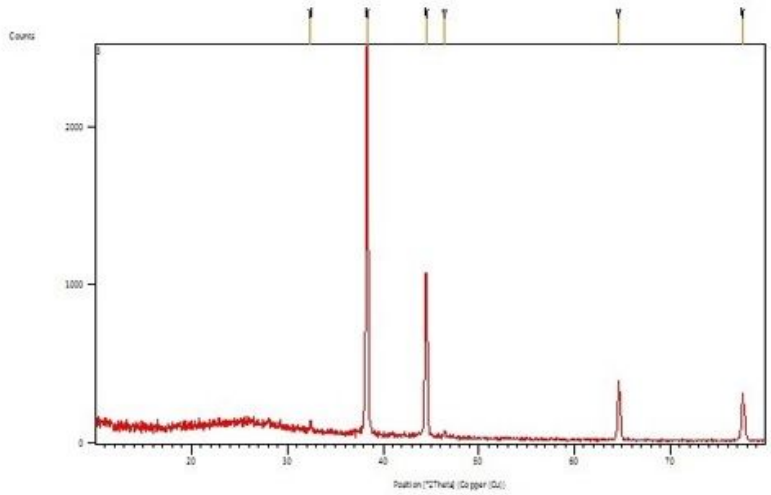

Figure 5

XRD patterns of silver nanoparticles

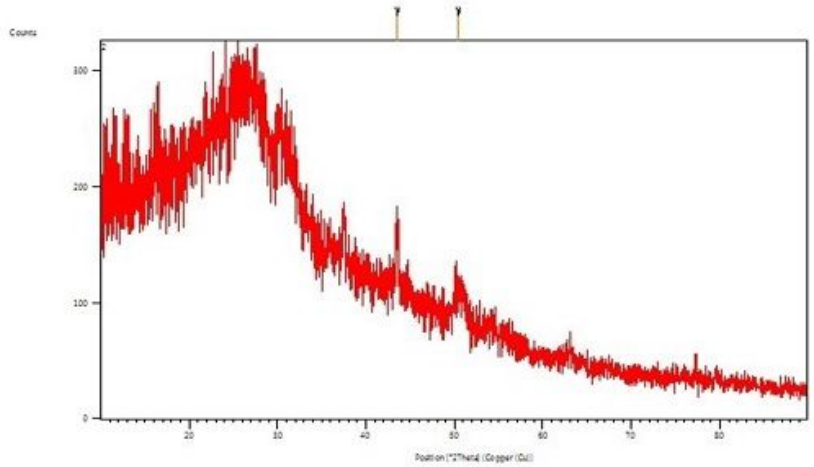

Figure 6

XRD patterns of nickel nanoparticles

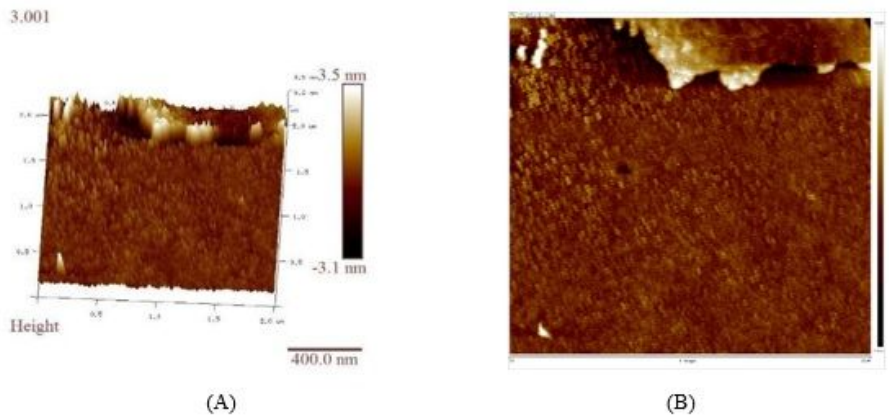

Figure 7

AFM image (A) 3D topography and (B) 2D topography of silver nanoparticles

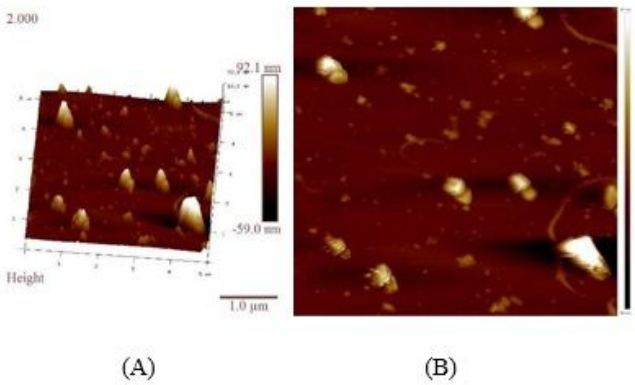

Figure 8

AFM image (A) 3D topography and (B) 2D topography of nickel nanoparticles

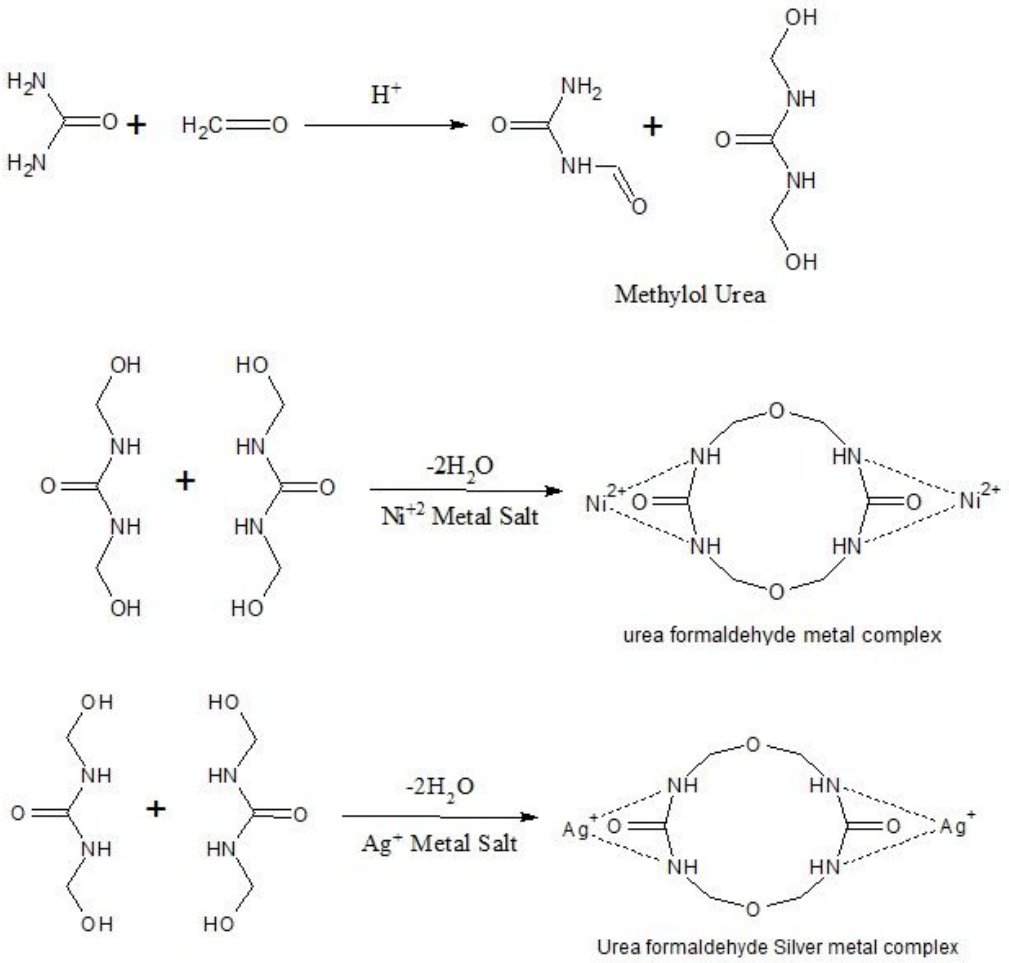

Figure 9

Reaction for the nickel and silver nanoparticle