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Effect of PVP, SDS and their concentration on the synthesis aggregated nano-nickel particles by hydrazine reduction.

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Abstract
The synthesis of nanosized nickel particles by chemical reduction of nickel chloride in a basic medium (pH10.2) was studied. Nano-sized agglomerated nickel particles also have been synthesized in the presence of polyvinylpyrrolidone (PVP), sodium-dodecyl sulphate (SDS) with their different concentration to study the effect of SDS, PVP and their concentration on the shape and size of the particles. Synthesized nano nickel particles were characterized by using SEM and TEM. SEM images showed the particles were spiky in morphology, some spherical shaped particles were also observed in the spiky structure.TEM characterization showed spherical nickel particles are 40-20 nm in diameter. The use of SDS and PVP produced nano-sized nickel particles which were much finer than where hydrazine was used on its own. PVP has shown some dispersion power, and was found to be capable of preventing nickel particles from gradual agglomeration.

Keywords: *Morphology, Reduction, Spiky, SDS, PVP, Hydrazine.*

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I. INTRODUCTION

Chemical reduction method has become a quite popular synthesis method to produce metal nanoparticles because of its relatively simple experimental procedure. Polyvinylpyrrolidone (PVP) has been widely used as a capping agent to control the growth of reduced nanoparticles. The PVP molecules reducing the surface energy and preventing a further agglomeration by attaching to the metallic nucleus. In several papers the changes in morphology and particle size of the produced nanoparticles were investigated as a function of the added amount of PVP. For instance, Pandey and Manivannan produced nickel nanoparticles by using nickel chloride and hydrazine hydrated of different molar ratio [1]. Silver nanoparticles synthesized by reducing silver nitrate as precursor, hydrazine hydrate as reducing agent, controlling the particle size by the PVP/$AgNO_3$ molar ratio [2]. The variation of amount of PVP in relation to other reagents was used, investigating the impact of the added amount of PVP on the size and morphology of synthesized Sn-based nanoparticles [3]. A study was performed to control the size and morphology of as-synthesized Ni-Sn nanoparticles by the amount of PVP added [4]. Nickel nano particles have found many applications in catalysts, batteries and superalloys. Size of the nano particles plays an important role in the field of catalysis. The finer the size of particle the catalytic activity will be higher. For example, in synthesizing optical active 3-hydrobutyric methyl ester, the yield rate has been found to be as high as to 85% if we use 30 nm nickel nanp particle as catalyst, and the reaction speed was also found to be 15 times more compared with that of normal nickel catalyst [5]. In preparation of porous metallic ensembles and as fillers for polymers they can also be used. The morphology and shape of the particles strongly effect the electrical and magnetic properties of the particles [6,7] Therefore, nickel nanoparticles with different morphologies and shapes, such as nanorod, nanospheres, hollow sphere, nanobelt and core–shell structure, have been synthesized by various methods [8,9]. In fact, nano-metre sized crystallites are often the primary product in precipitation from the solution. Such dispersions are unstable, and the so formed nanoscale crystal-lites become aggregated to form larger particles. Ni and coworkers have reported a chainlike nickel wire formed by self-assembly of small nickel crystallites in a soft templates [10]. Many different techniques have been used in the preparation of nanosized particles, for example, decomposition of carbonyls, the reduction of metal oxide salts [11], and reduction in solutions by strong reducing agents [12]. Reducing agents used include hydrazine, potassium borohydrides and sodium hypophosphite , which correspond to the formation of metal borides, metal phosphides [13] and pure metals. Recently, the use of surfactant–macromolecule clusters as template to prepare inorganic crystals has become an area of much research interest [14,15]. It is now well known that water soluble non-ionic macromolecule, such as polyvinylpirrolidone (PVP) or polyethyleneoxide interact strongly with

anionic surfactant, such as sodium dodecyl sulphate (SDS), in aqueous solution. Above the critical association concentration, surfactant aggregate on macromolecule chains in micellar structure, which is known as bound micelle [16]. In this research work, nanosized nickel particles/aggregates of 40 nm in diameter have been synthesized. Synthesis of nanosized NiCrAlY alloy powders (100 nm in diameter) for high temperature applications in aerospace and power generation industries in bulk quantities is the ultimate aim [17]. The currently used NiCrAlY alloy powder size is much bigger than 100 mm in diameter [18]. In this work, spherical/spiky spherical nickel nanoparticles with diameter,40nm have been successfully synthesized with PVP and SDS being used separately . Morphology, composition, crystallite size and formation of nickel nanoparticles were investigated using scanning electron microscope (SEM) and high resolution transmission electron microscope (HRTEM)

II. EXPERIMENTAL

In this research we used nickel chloride analytical grade, SDS (LOBA Chemie, Mumbai, India), PVP (Winlab, Market Harborough, UK), hydrazine hydrate solution (LOBA Chemie) and sodium carbonate (Sigma-Aldrich, St Louis, MO, USA); distilled water was used in the preparation of all the solutions. In this research a thermostatically controlled hot plate with magnetic stirrer (yellow line) was used. 10 g of NiCl2 dissolved in a glass beaker containing 100 mL of deionized water, temperature was maintained at 400C. The pH of the solution was maintained at 10.2 by adding concentrated solution of sodium carbonate. 150 mL of hydrazine slowly added to the solution while stirring all the time. Temperature of the solution was increased to 600C before adding the hydrazine. At 600C formation of grey/black precipitates in the beaker meant that nickel particles have started to form. This reaction is not instantaneous and can take several hours for completion. At 600C when the nickel nano particles formed, there is an expansion in volume of the reacting solution, care had to be taken not to spill the solution over the reacting vessel, e.g. much bigger beakers were used that could handle the volume expansion of the reacting chemicals. As the reaction proceeded, the top of the beaker became covered with thick froth; simultaneously, nickel particles were deposited at the bottom of the reacting vessel. The grey/black particles so formed were the aggregated nano-sized nickel particles. After collection of the product they were centrifuged (4000 rev min), washed with distilled water and ethanol for three times and desiccated at room temperature before characterization. The froth also contained a fair amount of fine nickel particles and these particles were recovered by washing with acetone and water before drying. Experiments were carried out using different amount of PVP and SDS to study the effect of PVP, SDS and their concentration on the morphology and size of the nickel nano particles. The nickel particles were characterized using SEM and TEM.

II. RESULT AND DISCUSSION

Size and morphology of the nickel nano-particles plays very important rule on its applications. Effect of PVP, SDS and their concentration on the size and shape of the particles were studied in this research. Figures 1 is the SEM image of nickel nano-particles synthesized using hydrazine only without any SDS and PVP, figure shows that the shape of the particles seems to be spherical. Figures 2 is the SEM image of the nickel nano-particles synthesized using hydrazine with $7gL^{-1}$ PVP without any SDS, figure shows that the shape of the particles are spiky. Figure 3 is the SEM image of nickel nano-particles synthesized using hydrazine with $10.5gL^{-1}$ PVP without any SDS, image shows that the synthesized particles shape is spiky but there are a lot of small particles are observed. Figure 4 is the TEM image of the nano-nickel particles synthesized using hydrazine only without any SDS and PVP, image shows that nickel nano-particles are around 45 nm in diameter. Figure 5 is the TEM image of the nano-nickel particles synthesized using hydrazine with $7gL^{-1}$ PVP without any SDS, image shows that the synthesized particles are 30 nm in diameter. Figure 6 is the TEM image of the nano-nickel particles synthesized using hydrazine with $10.5gL^{-1}$ PVP without any SDS, image show that the synthesized particles are 10 nm in diameter.

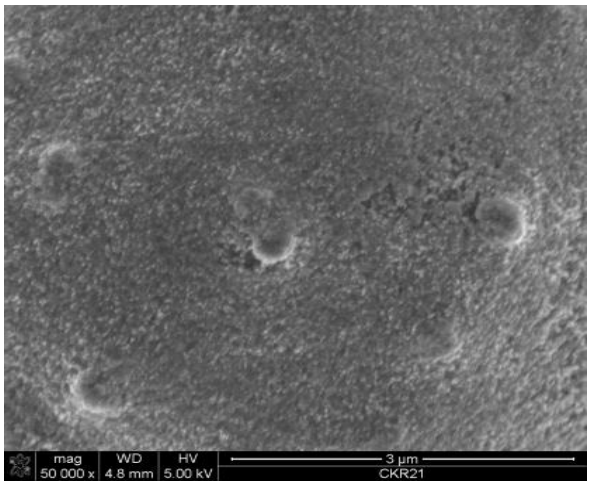

Figure 1

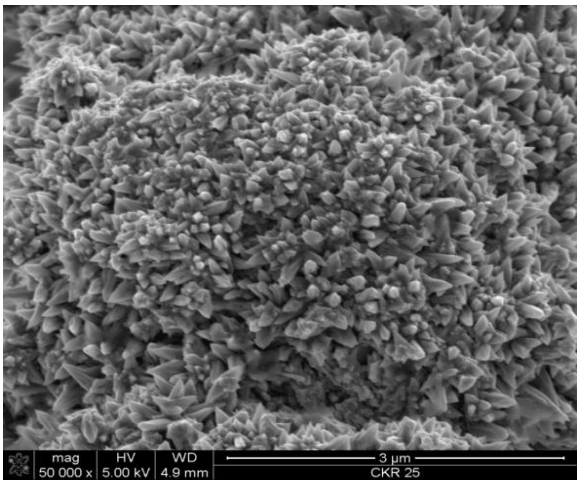

Figure 2

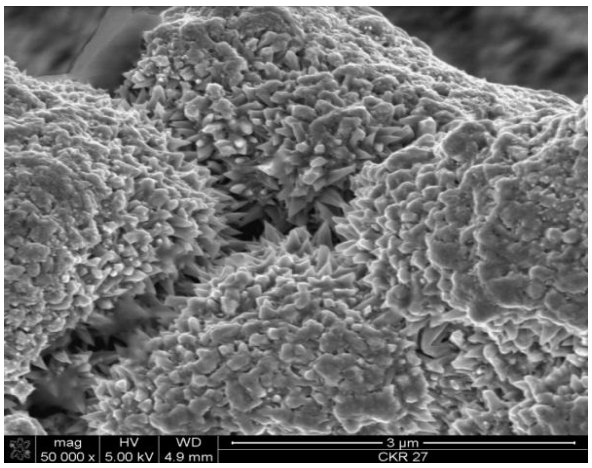

Figure 3

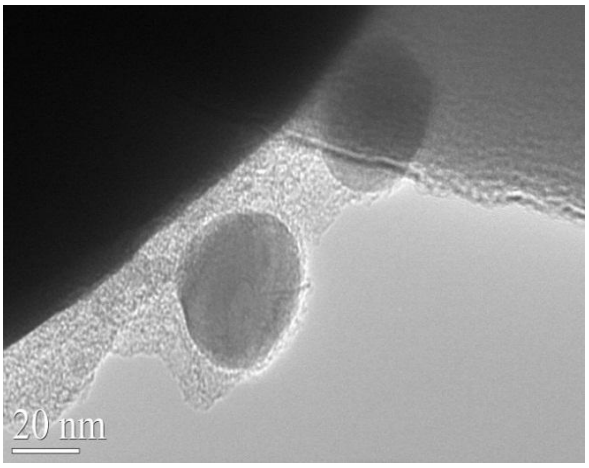

Figure 4

From the above results we can say that PVP has the capacity to retain the size of the particles, presence of PVP molecule decrease the size of the synthesized nickel nano-particles. Increasing the concentration of PVP particle size decreases. The morphology of the particles changes from spherical to spiky increasing the concentration of PVP. Figures 7 is the SEM image of the nano-nickel particles synthesized by reducing nickel chloride with hydrazine in the presence of 0.288g SDS without any PVP, image shows that the synthesized particles are spiky but we can observe many spherical tiny particles. Figure 8 is the SEM image of the nano-nickel particles synthesized by reducing nickelchloride with hydrazine in the presence of 0.576g SDS without any PVP, the particles have spiky morphology but the spikiness becoming sharper. Figure 9 is the SEM image of the nano-nickel particles synthesized by reducing nickelchloride with hydrazine in the presence of 1.15g SDS without any PVP, particles are spiky but there are also many spherical particles. Figure10 is the TEM image of the nano-nickel particles synthesized by reducing nickel chloride with hydrazine in the presence of 0.288g SDS without any PVP, image shows that synthesized nano-nickel particles are around 25 nm in diameter. Figure11 is the TEM image of the nano-nickel particles synthesized by reducing nickel chloride with hydrazine in the presence of 0.576g SDS without PVP, image shows that synthesized nano-nickel particles are less than 10 nm in diameter. Figure12 is the TEM image of the nano-nickel particles synthesized by reducing nickel chloride with hydrazine in the presence of1.15g SDS without any PVP, image shows that synthesized nano-nickel particles are around 20 nm in diameter.

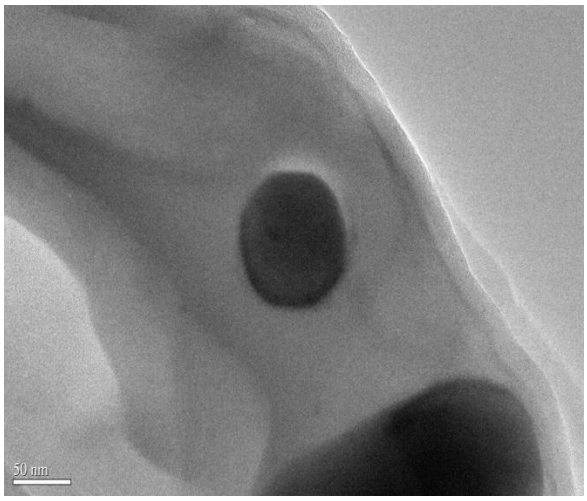

Figure 5

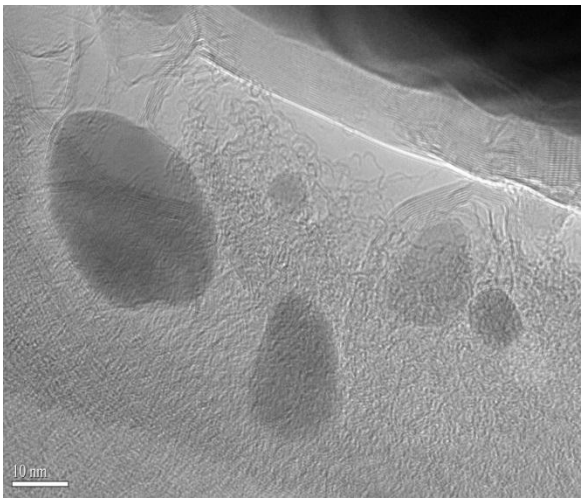

Figure 6

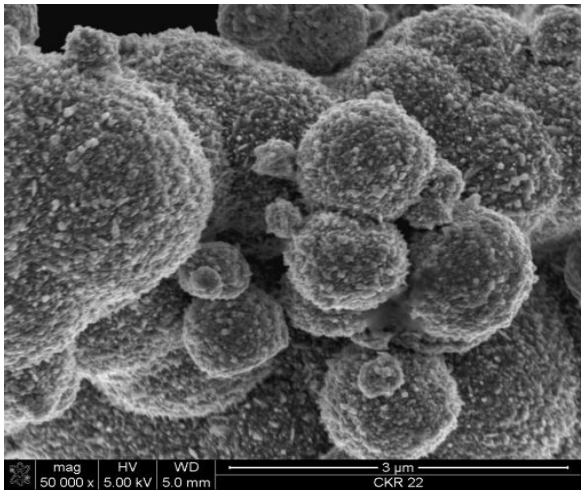

Figure 7

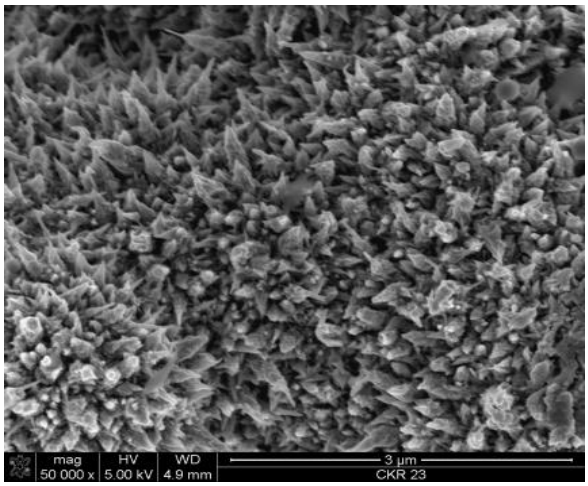

Figure 8

Effect of surfactant molecule (SDS) on the size and shape of the synthesized nano-nickel particles is that presence of SDS in the reaction mixture can produce more finer particles than the particle synthesized without any SDS. Also increasing the concentration of the SDS the particle size decreases gradually. But up to a certain concentration this trend observed, when the concentration of SDS is1.15mmolL^{-1} particle size again start to increase. This is because we know that surfactant form micelle in which the newly born nano-nickel particles enter. Figure 13 is the formation of micelle by the SDS molecule which effect the morphology of the synthesized nickel nano particles.

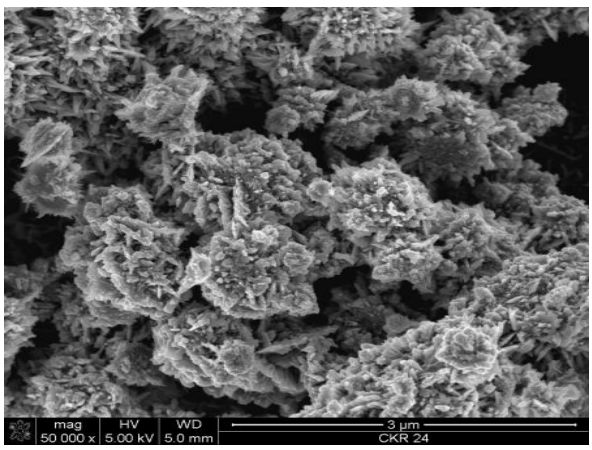

Figure 9

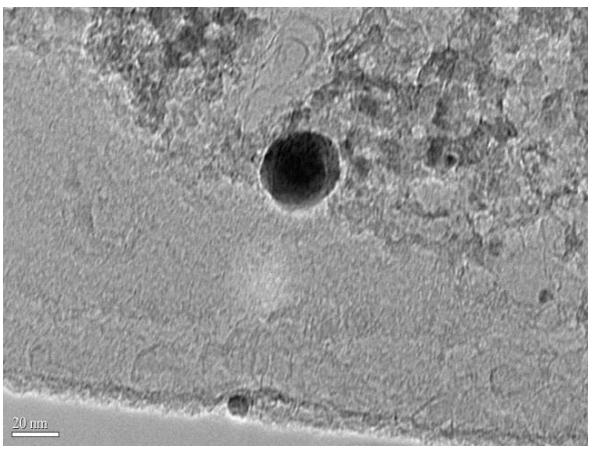

Figure 10

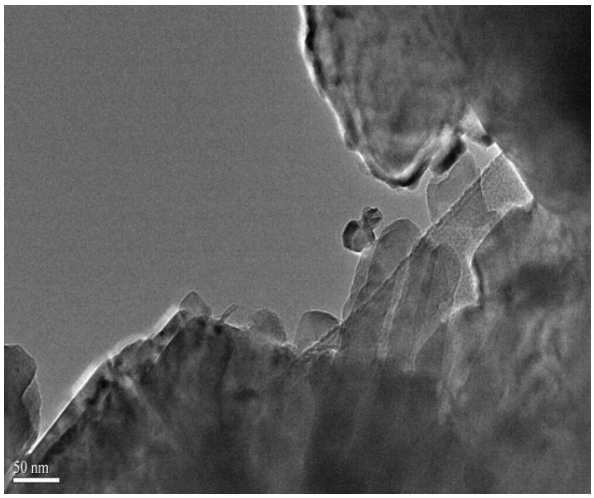

Figure 11

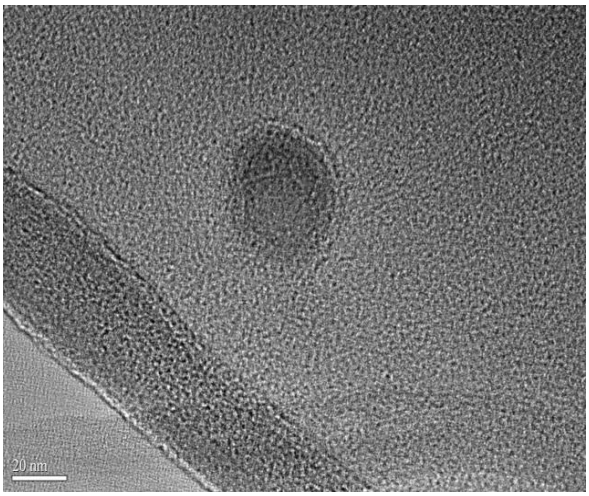

Figure 12

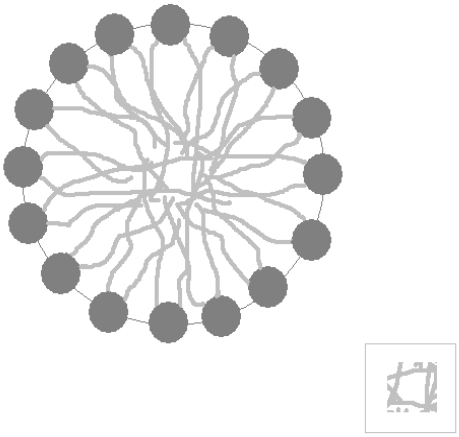

Figure 13

III. CONCLUSION

Aggregated nano-nickel particles have been synthesized by a chemical reduction of nickel chloride using hydrazine as reducing agent in a strong basic medium at pH 10.2. A simple cationic polymer PVP and anionic surfactant SDS was used in different concentration to study the effect of cationic polymer and anionic surfactant on the shape and size of nickel nano particles. Our research shows that the presence of PVP and SDS affect the shape or morphology and size of the particles. Presence of PVP or SDS reduces the size of the particles and increasing the concentration of PVP and SDS the size of the particles also decreases. Morphology of the particles changes from spherical to spiky in presence of PVP and SDS.

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