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Decolourization of C. I. Reactive Black 5 by PVP stabilized Nickel nanoparticles

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Abstract- C.I. Reactive Black 5 dye was decolourized by Nickel nanoparticles with Polyvinyl pyrrolidone (PVP) as capping agent. The characterization of nanoparticles was done by transmission electron microscopy (TEM) and by Dynamic Light Scattering (DLS). The effect of pH, dye concentration, nanoparticles concentration, alkali addition, salt addition and duration of treatment on dye decolourization was studied. FT-IR analysis of the degradation products revealed the attachment of some amount of degraded product of dyes on the nanoparticles. The color removal efficiency and COD reduction were obtained as 98.9% and 56%, respectively.

Index Terms— C. I. Reactive Black 5, Decolourization, Nickel nanoparticle, polyvinyl pyrrolidone.

I. INTRODUCTION

Wastewater from many industries, such as textile, leather, food processing, cosmetics, and dye manufacturing contain various amounts of dyes which are water pollutants. They represent a relatively large group of organic chemicals, not biodegradable, with a potential toxicity for the aquatic ecosystem. When they are present even in moderate concentrations (10–200 mg/L), their presence can significantly affect photosynthesis activity and reduce light penetration, thus affecting the growth of plants and impacting invertebrate and other forms of wildlife. Dyes can cause allergy, dermatitis, skin irritation, cancer, and mutations in humans [1], [2]. Also, some dyes, such as benzidine, were identified as carcinogens that may be reformed as a result of metabolism.[3] Consequently, the dyes must be removed from the industrial effluents before their discharge in the environment or in the municipal waters.

Many processes are employed to remove dye molecules from colored effluents, and the treatment methods can be divided into three categories: (i) physical methods such as adsorption [4]-[6], membrane filtration [7], and ion exchange [8]; (ii) chemical methods such as chemical oxidation [9], electrochemical degradation [10], and ozonation [11]; and (iii) biological degradation [12]. The main disadvantage of physical methods is that they simply transfer the dye molecules to another phase rather than destroying them, and they are effective only when the effluent volume is small [13]. Chemical methods with high cost are rarely used in the actual treatment process, and the disposal of sludge containing chemicals at the end of treatment requires further use of chemicals [14], [15]. Biological degradation processes suffer from low degradation efficiency or even no degradation for some dyes [16].

Metal nanoparticles used for decolourization, here nickel have attracted much attention because of their use in numerous practical applications, such as magnetic storage materials [17], magnetic inks [18] and catalysts [19]. Numerous physical and chemical methods have been used to produce high-quality nickel nanoparticles, such as metal evaporation-condensation [20], electrochemical methods [21], metal salt reduction [22], ultrasound irradiation [23], ultrasound spray pyrolysis [24], polyol methods [25] and neutral organometallic precursor decomposition [26], [27]. Generally, chemical reduction methods have the advantage of simplicity, precise control of the size and low cost compared with physical methods. For controlling the size and shape of the nanoparticles, various physical and chemical synthetic routes were developed. An important feature in the production of nanoparticles is the ability to keep them physically separated from each other preventing irreversible aggregation especially in magnetic nanoparticles which tends to aggregate due to magnetic dipole-dipole attractions to reduce their surface energy. The aggregation of nanoparticles can be eliminated by steric (coating the particles with a surfactant layer) or ionic (inducing opposite charge on the particle surface) repulsion [28]-[30]. Polymers such as dextran or polyethylene glycol, PVP, low molecular weight compounds such as citric or oleic acid or inorganic materials such as silica or gold have been reported to coat the magnetic nanoparticles in order to prevent irreversible aggregation. The surface coating not only protects the particles from aggregation, but it can also affect their size and polydispersity, hydraulic diameter, shape and the total effective moment of the nanoparticles [31]-[34]. However, to obtain non-agglomerated nanoparticles with a well-controlled mean size and a narrow size distribution is still a challenge.

In this work successful approach has been sought for the above problem of agglomeration. A feasibility study for removing colour of dye solution using chemically synthesized monodisperse nickel nanoparticles from nickel salts



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in a non-aqueous solution using PVP as a stabilizer was made in terms of the experimental parameters such as time, nanoparticle concentration, initial dye concentration, and pH. Also the effect of presence of salt and alkali in the dye solution and COD reduction was investigated.

II. MATERIAL AND METHOD

Chemicals

C. I. Reactive Black 5 was purchased from Colourtex Industries Limited, Mumbai. Nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, $\geq 98\%$), Hydrazine hydrate ($\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$, Mw 50.06), Acetone (Mw 58.08, 99%), Polyvinyl pyrrolidone (PVP K-30), Sodium hydroxide (NaOH, MW approx. 40), Acetic acid, Ethanol (99.7%), Chloroform, Methanol, Soda Ash and Glauber salt were supplied by S D Fine-Chem Limited (SDFCL, Mumbai).

Synthesis of Ni-PVP nanoparticles

A 3.3mM solution of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ in 200 ml of ethanol was added to a 500 ml round bottom flask at room temperature. 0.125 gram of PVP was then added to this solution and the mixture was stirred until the total dissolution of PVP. 32 ml of hydrazine hydrate followed by 6.8ml of 1M NaOH solution was added under strong magnetic stirring. After this, the solution was heated at 60°C with constant stirring and within 5 minutes the solution turned black indicating the formation of nickel nanoparticles. During synthesis no additional nitrogen atmosphere was used, as hydrazine itself releases N_2 gas on its decomposition. The solution was then allowed to cool in ambient conditions at room temperature. After cooling, acetone was added in order to precipitate out the nanoparticles. The resulting solution was vacuum filtered and particles were collected and washed several times with a chloroform-methanol (1:1) solution. Finally, the nanoparticles were dried in vacuum dryer at 50°C and collected for further studies. Similarly, synthesis of Ni nanoparticles without PVP addition was also done. [35].

Characterization of nanoparticles

The size and morphology of the nanoparticles were estimated using transmission electron microscope (TEM) (Model CM 200, Philips) operated at an accelerating voltage of 200 kV. Dynamic Light Scattering (DLS) was done to determine the mean diameter of the nanoparticles (Malvern model-v2.1).

Batch decolourization studies

C. I. Reactive Black 5 used in this study is of commercial grade and widely used in textile industry. The purification of the dye was not done to give a practical significance to the study. Dye structure is shown in Fig 1. Different concentration of the dye stock solutions were prepared in deionized water and decolourization experiments were performed in an open batch system at room temperature, $28 \pm 2^\circ\text{C}$. To keep nanoparticles suspended, dye stock solutions were stirred using shaker machine (Rossari Labtech, Mumbai) at 70 rpm. From the top suspended solution, samples were withdrawn at fixed intervals of time, centrifuged for 5 minutes at 3000 rpm. Centrifuged solution was then pipetted out and the decolourization rate was monitored using UV-VIS spectrophotometer (UV-VIS 8500, TECHCOMP Limited, Hong Kong.) at 597.3 nm wavelength. The percentage of dye decolourization was found out by first preparing the calibration curve of the dye. COD of the treated effluent was determined according to ISO 6060(1989) on Levibond MD200 COD VAR10. Degradation products on the nanoparticles was analyzed by FT-IR 8400S (CE), Shimadzu, Japan.

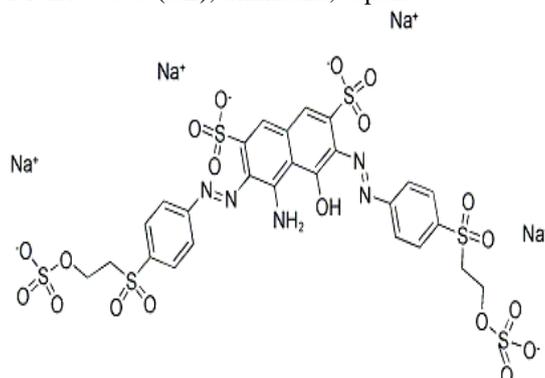


Fig. 1 Chemical structure of C. I. Reactive Black 5

Nanoparticle characterization

TEM analysis of Ni-nanoparticles is shown in Fig. 2. The Ni-nanoparticles without PVP are spherical in nature having size 29-243 nm and agglomerate in a self-assembled flower like structures, Fig. 2(a). Whereas Ni-PVP nanoparticles have size in the range of 20-44 nm which were dispersed within the PVP matrix, as shown in Fig. 2(b). The particle size distribution Ni-PVP nanoparticles determined on particle size analyser shows that the particle size of 55 nm (Fig. 3).

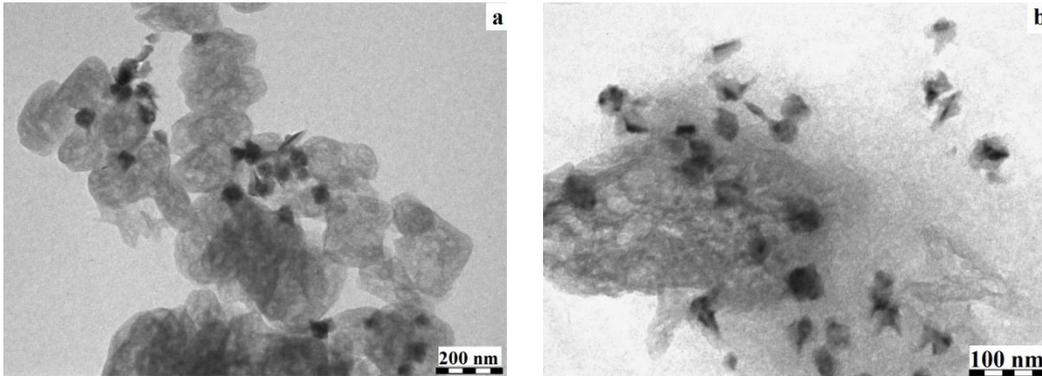


Fig. 2 TEM image of Ni-nanoparticles (a) without PVP and (b) with PVP

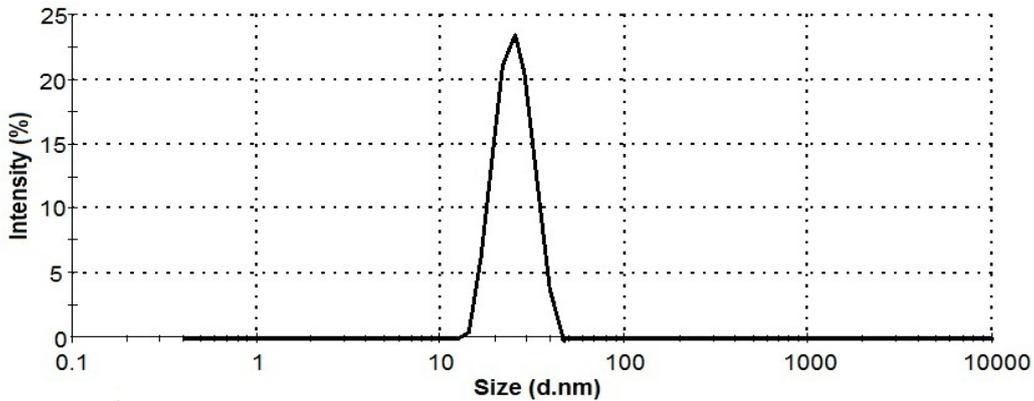


Fig.3 Particle size analysis for Ni-PVP nanoparticles

Dye decolourization

Fig. 4 describes the effect of time on % decolourization of dye (100 mgpl) with different nanoparticles concentration. From the plot we can see that as the concentration of nanoparticles and time of treatments increases, % decolourization also increases.

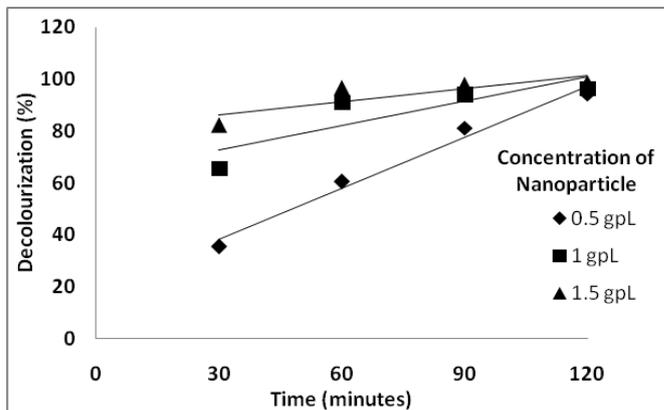


Fig. 4 Effect of time on % Decolourization of dye (100 mgpl) with nanoparticles concentration

Effect of initial dye concentration

Fig. 5 show that, as the initial dye concentration taken increases the decolourization efficiency decreases for the same amount of nanoparticles concentration. For further study we had taken 100 mgpl of dye.

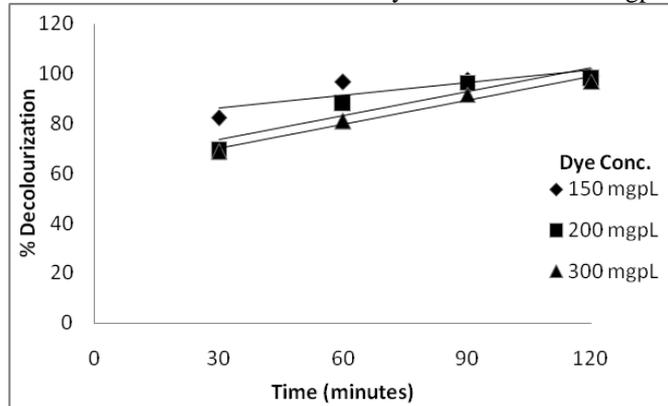


Fig. 5 Effect of Dye concentration on % Decolourization using nanoparticles (1.5 gpl) for 120 minutes

Effect of pH

The pH of the aqueous solution is a complex parameter which is related to the ionization state of the nanoparticles surface and that of the reactants and products such as acids and amines present in the solution. Dye degradation was evaluated in a pH range of 2-7 and is shown in Fig. 6. It can be seen that maximum % decolourization was observed at pH 7 (98.93% in 120 min) [36]. In alkaline pH, the metal nanoparticles form oxides which occupy its surface. Hence their activity gets reduced and decolourization is not observed in alkaline pH.

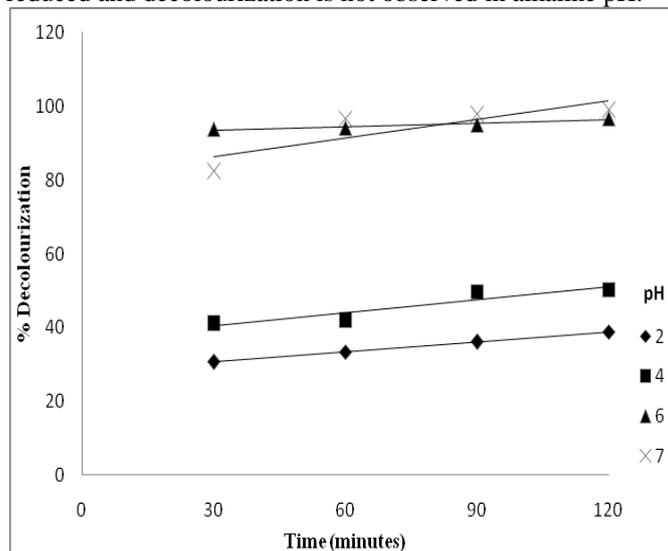


Fig. 6 Effect of pH variation on % Decolouration with nanoparticles [1.5 gpl] and dye [100 mgpl] for 120 minutes

Effect of alkali

Since the reactive dye taken for the study is always applied on the substrate in presence of salt and alkali, we investigated the effect of presence of these two chemicals on the efficiency of nanoparticles on decolourization. To study the effect of alkali, we added 20 gpl of soda ash in the dye solution which increase the pH of solution to pH 11.3. The pH was brought down to the value of pH 7 by addition of acetic acid and then % decolourization study was done. From Fig. 7 we can see that when soda ash is added in dye solution the % decolourization by nanoparticles gets reduce. On bringing pH to basic condition, the decolourization rate by nano particles was found to be decelerated because the oxide precipitated onto the surface occupying the active sites, soon terminating the reaction [37].

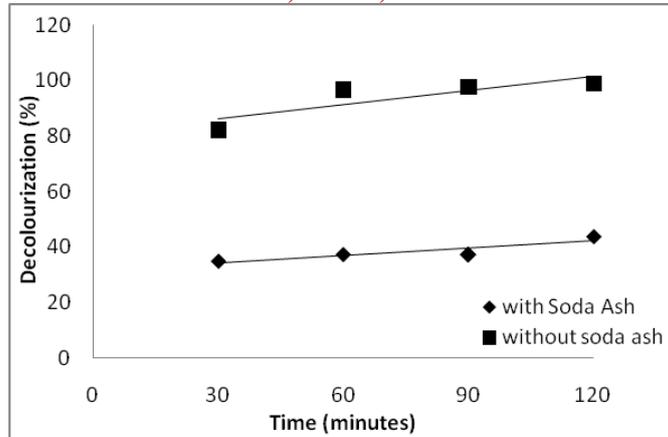


Fig. 7 Effect of Soda Ash (20 gpl) addition on % Decolourization with nanoparticles (1.5 gpl) and dye (100 mgpl)

Effect of salt and alkali

Effect of soda ash and glauber salt addition on % decolourization by nanoparticles is shown in Fig. 8. We can observe that % decolourization efficiency gets reduced when both soda ash and glauber salt were present in the dye solution. This could be because of oxide formation on the surface of these nano particles reducing its reductions capacity.

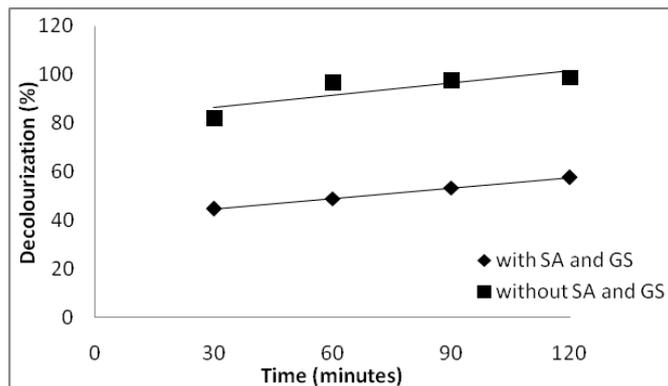


Fig. 8 Effect of Soda Ash (SA) 20 gpl and Glauber Salt (GS) 40 gpl addition on % decolouration with nanoparticles (1.5 gpl) and dye (100 mgpl)

Efficiency of the recycled nanoparticles

To investigate the economy of the reductive degradation process, which is a measure of the number of times a nanoparticle can be reused without sacrificing its efficiency, the life span of Ni-PVP nanoparticles was tested. After the first degradation cycle, the treated solution was kept standing for a fixed time duration and then the supernatant was decanted. The nanoparticles were washed thoroughly with distilled water and reused for degradation of a fresh dye solution. All experiments were performed at neutral pH optimized by study and dye concentration used was 100 mgpl for 120 minutes. The results are presented in Fig. 9. Decolourization for the first cycle was 98.94 % where as when repeatedly used for twelfth cycle it came down to 89.15 %. Thus after twelfth cycle the decrease in decolorization efficiency was approximately 10 % as compared to fresh. It was found that the reactivity of the nanoparticles decreased as the time progressed. It is very well understood that metal corrosion in water is more rapid than in air, due to the presence of hydroxyl ions and dissolved oxygen, which can attack the bulk metal and form metal hydroxides and oxides on its surface. The hydroxides are easily leached out from the surface and this exposes the inner layer of core metal for further attacks, resulting in loss of the metallic ion and reduction in the nanoparticle efficiency for decolourization. Though, the presence of stabilizer PVP prevents the formation of metal oxide layer which slows reduction in the efficiency. [36]

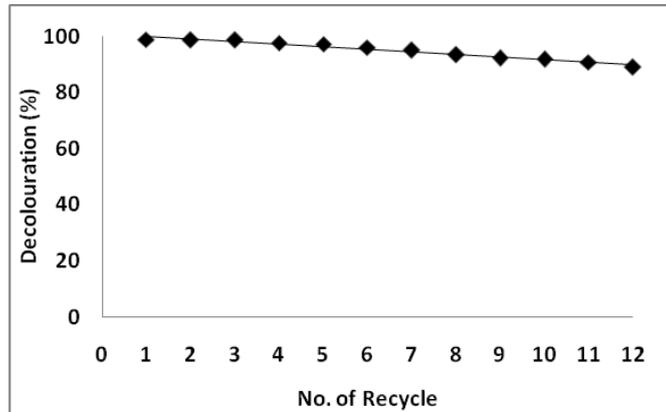


Fig. 9 Efficiency of the recycled nanoparticles

FTIR measurements

120 minutes after the decolourization reaction, particulate materials were collected by centrifugation and removed from the supernatant solution. The collected particles were vacuum-dried at 60°C for 6 hours and characterized by FT-IR spectroscopy. By FT-IR technique we can identify groups of the degraded product of dyes on the Ni-PVP nanoparticles surface. The infrared spectra of nanoparticles before and after the decolourization process were observed in the range of 4000–400 cm⁻¹ (Fig. 10). When compared with the original Ni-PVP nanoparticles, some new bands were found on the Ni-PVP nanoparticles after decolourization including the vibrations at 3282 cm⁻¹ (O–H bending), 1604 cm⁻¹ and 1566 cm⁻¹ (–N–H– bending), 1315 cm⁻¹ (–CH₂– bending), 1166 cm⁻¹ (–SO₃ bending), 979 cm⁻¹ (–C–H– bending), respectively. The above findings suggest that some amount of degraded product gets attached on the surface of the nanoparticles [38].

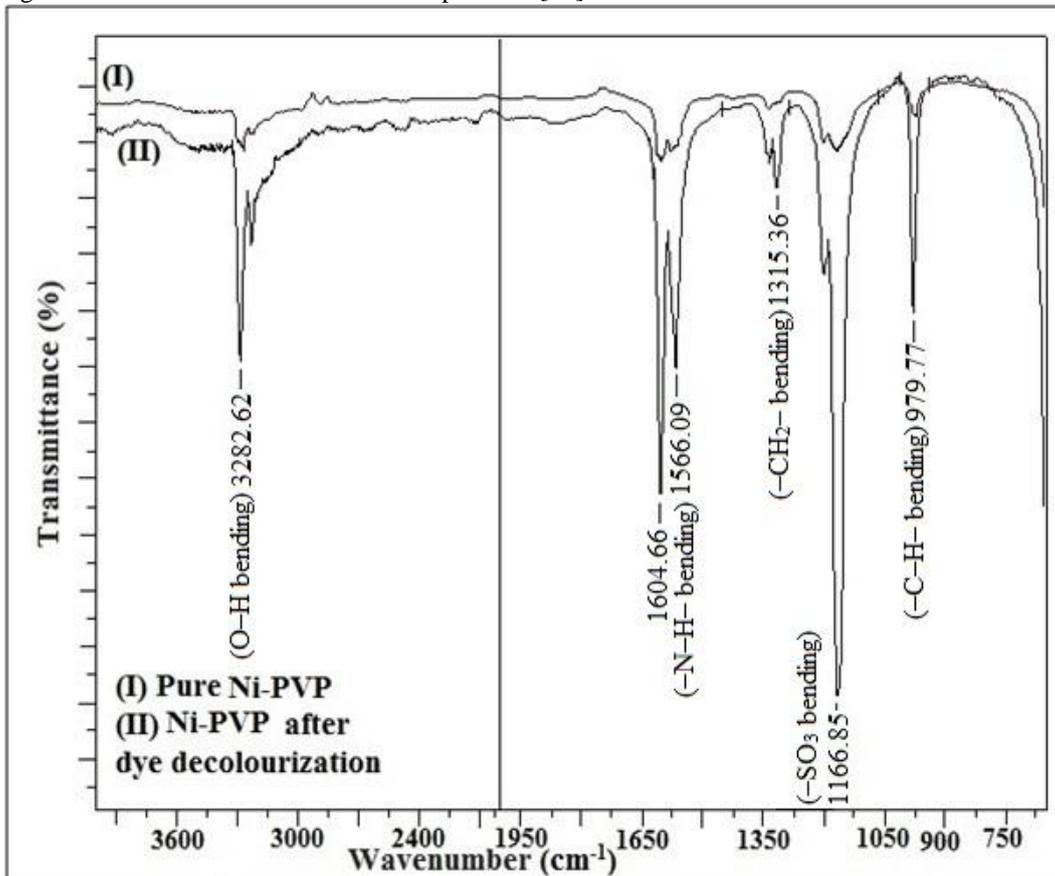


Fig.10 FTIR spectra of nanoparticle and dye-loaded nanoparticle sample



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COD reduction

Dye concentration results reported previously in the study were based on the spectrophotometric analysis of the dye solutions. But the spectrophotometric evaluation cannot be interrelated to reduction of COD [39]. Hence the evaluation of reduction in COD was performed. COD measurement experiments were conducted with 100 mgpl initial concentration of dye and 1.5 gpl of Ni-PVP nanoparticles. The initial COD of the dye was found to be 100ppm of the stock dye solution and the value gets reduced up to 44ppm after complete decolourization. So, it can be inferred that Ni-PVP nanoparticles not only removes the color from the effluent but also reduces the COD from the treated effluent.

IV. CONCLUSION

The work carried out put forth a novel concept of decolourization of dye effluent using nickel nanoparticles. The usage of PVP in the synthesis of nanoparticles gives a better dispersion and reduces the agglomeration tendency. Also the reduction of COD is achieved along with decolourization. The nanoparticles can be recycled by simple separation method and can be reused almost 10 to 12 times with very low reduction in their decolourization efficiency. The generation of sludge through the usage of these nanoparticles is minimal as compared to the conventional methods used for decolourization such as coagulation and flocculation.

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