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Coating of Polyvinylpyrrolidone on Biosynthesized Iron Oxide Nanoparticles

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Abstract: In the recent study, nano-particles of iron oxide are gaining significance because these have vital properties and their multi-functional utilizations like medical diagnostic, catalytic degradation, hazardous waste treatment and biomedical applications. There are many methods for preparation of nanoparticles. But green synthesis is gaining attraction due to its non-toxicity and biocompatibility. In this study, iron oxide nanoparticles were synthesized from leaves of *Camellia sinensis* (green tea) and ferric chloride solution at 50°C temperature. After formation of iron oxide nanoparticles polyvinylpyrrolidone was coated on them (during synthesis and after synthesis of nano-particles). Plant extract contains different types of polyphenols which play major role in reduction and stabilization of nano-particles along with polyvinylpyrrolidone. Characteristics of synthesized nanoparticles were determined by different techniques. By dynamic light scattering technique size of nanoparticles was measured which was 85nm, 120nm and 133nm. UV-Visible technique exhibited that nanoparticles showed maximum absorption at 280nm. FTIR revealed about different function groups. EDX analysis found out about elemental composition and atomic percentage of polyvinyl pyrrolidone coated iron oxide nanoparticles which were carbon, oxygen, iron and silicon and their percentage was 64.39%, 34.40%, 0.12% and 0.05% respectively. Scanning electron microscopy showed that coated and uncoated iron oxide nanoparticles had size 10 µm and 50 µm respectively. XRD determined the crystal structure and size of iron oxide coated with polyvinyl pyrrolidone (dry and wet method) was 42.58nm and 28.69nm respectively.

Keywords: Green Synthesis, PVP, Nanoparticles of Iron Oxide

1. Introduction

Nanoparticles have size in the range between 1-100nm. These particles have great attraction due to their special properties like large surface area and high reactivity as compared to larger molecules [1].

These are synthesized by many ways like physical synthesis, chemical synthesis and bio synthesis. First two methods are adverse because these methods cause adverse effect on the environment. But green synthesis approach is relatively new, cost effective and ecofriendly. It is executed at atmospheric temperature and pressure. This method is free of precarious agents and toxic by-products. Main edge of this technique is to use bio-renewable sources like fruit peels or green leaves [2].

Camellia sinensis (green tea) leaves contain alkaloids (caffeine, theobromine, theophylline, etc.), flavonoids (has anti-inflammatory, antioxidant, anti-microbial effects), polyphenols (catechins of gallocatechin, epicatechin). These catechins exhibits anti hypercholesterolemia activity, maintains elasticity of the skin, strengthens the capillaries etc. Iron nanoparticles synthesized from green tea leaves used in biomedicine, drug delivery and disease detection. These nanoparticles are also used in the treatment of tumor cells [3].

Polyvinylpyrrolidone (PVP) is a water soluble and non-toxic polymer which acts as a binding agent in many pharmaceutical tablets. It is favored due to its low cost and easy availability. It is an excellent candidate for surface modification of nanoparticles.

Iron oxide nanoparticles exhibit colloidal properties in water with negatively charged surface. These nanoparticles must be stabilized for drug delivery to increase efficiency. Iron oxide nanoparticles are coated with PVP to obtain stability. The interaction of iron oxide nanoparticles and PVP is determined by zeta potential values with different polymer concentrations [4].

1.1. Approaches for Synthesis of Nanoparticles

Following are two general approaches for the synthesis of nanoparticles:

I. Top-down approach

In the top-down approach, nano-structures are manufactured from substance by using different methods like laser ablation, etching mechanical milling and sputtering. Even though by using these techniques we can get best and pure nanoparticles but these methods are not suitable economically and equipment is used in it. [11]

II. Bottom Up Approach:

In this approach, precursors can be used in any phase like solid, liquid, or gas to form nanoparticles. Usually metal nanoparticles are formed by this approach. These procedures can be carried out in atomic condensation, sol–gel phases, aerosol processes, vapor deposition, thermal decomposition, co-precipitation, and by biosynthesis.

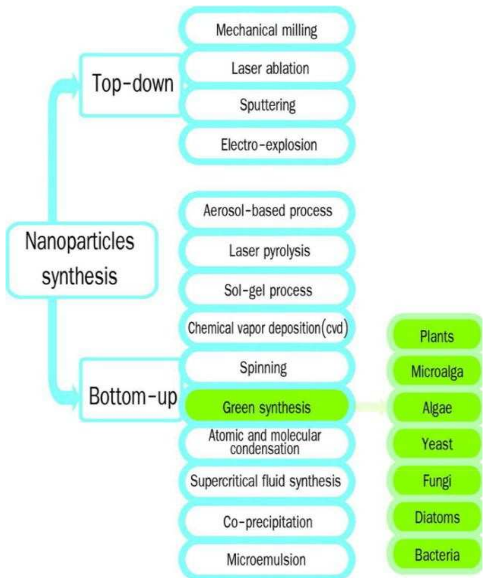


Figure 1. *Top Down and Bottom Up Approaches for Synthesis of Nanoparticles [12].*

1.2. Green Synthesis of Nanoparticles

In the green synthesis, plant extract and metal salt is used. Plant extract contains different phytochemicals like alkaloids, phenolic acid and proteins that behave as a reducing or capping agent while metal salt is used as a precursor. For example for the synthesis of iron nanoparticles different salts of iron such as $FeCl_3 \cdot 6H_2O$, $FeCl_2 \cdot 4H_2O$, $FeSO_4$ or $Fe(NO_3)_3$ are used as an iron precursor. In this strategy reaction completes at room temperature and voluntary. It takes few minutes to complete the reaction. It is an eco-friendly and low cost technique [5].

2. Experimental Work

2.1. Plant Material

To prepare the iron oxide nanoparticles leaves of *Camellia sinensis*were selected. *Camellia sinensis*is the specie of family Theaceae. It was purchased from the local market of Lahore.

2.2. Preparation of Iron Chloride Solutions

For the manufacturing of nano-particles, 0.05 molar iron chloride solution was formed by dissolving 8.1gram of ferric chloride in 1 liter distilled water.

2.3. Preparation of Extract

20g of green tea leaves were weighed and washed carefully two to three times with running water and then with distilled water to pull out surface contaminations. After this washed green tea leaves and 1000 ml distilled water were added in beaker and heated for 40 minutes at 80°C in water bath. The extract obtained was filtered by Whatmann No. 1 filter paper. Extract was bright orange in color.

2.4. Formation of Nano-Particles

These were formed by using bio reduction technique with aqueous solution of 0.05molar iron chloride. The filtered extract of green tea leaves was mixed with $FeCl_3$ solution in 2:1 ratios respectively. Add iron chloride solution drop by drop into green tea leaves extract and placed it on magnetic stirrer. There is an immediate change in color on adding iron chloride solution, from bright orange to black. This is the indications of synthesis of nano- particles. After completion of reaction, stirred the solution for 30 minutes at magnetic stirrer. After this, the solution was filtered by using membrane filter paper of 0.2 micrometer in size. Then centrifuge the extract at 10000rpm for 20 minutes for the formation of pellet. Then found pellet of iron oxide nanoparticles was washed two times by distilled water and one time by ethanol. Then again centrifuge them and dry by the help of acetone. After that dried iron oxide nanoparticles were obtained.

2.5. Coating of Polyvinyl Pyrrolidone After Synthesis of Iron Oxide Nanoparticles

For coating, 0.5g of iron oxide nanoparticle was added in 60ml of distilled water and sonicated for 4 hours to prevent the agglomeration. After the sonication 1.25 g of polyvinyl pyrrolidone was added and was set on shaker for twenty four hours. Afterwards, solution was centrifuged at 10000 rpm for 20 minutes to form the pellet of nanoparticles. Then washing is done by distilled water, ethanol and acetone respectively and dried at room temperature for 24hours.

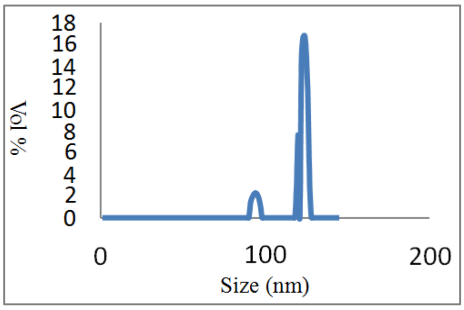
2.6. Coating of Polyvinyl Pyrrolidone During the Formation of Nano-Particles (Wet Method)

The nano-particles were biosynthesized by utilizing 0.05 M solution of $FeCl_3$ and green tea extract. For coating 1.25g of polyvinyl pyrrolidone was dissolved in 250 mL green tea extract. After adding polyvinyl pyrrolidone color of green tea extract changed from bright orange to dull orange. Solution of green tea extract and polyvinyl pyrrolidone mixed drop by drop with $FeCl_3$ solution in 2:1 ratios and kept on magnetic stirrer at room temperature. By adding $FeCl_3$ the color of solution (green tea extract and polyvinyl pyrrolidone) changed from dull orange to black that was the indication of the synthesis of nano-particles. Solution of nano- particles was filtered by utilizing 0.2 micrometer filter paper to separate large size nanoparticles from small size nanoparticles. The filtrate was centrifuged at 10000 rpm for twenty minutes to make pellet of nanoparticles. Washing of nanoparticles done by water and alcohol to remove the contamination from nanoparticles. Then nanoparticles were dried by using acetone at room temperature.

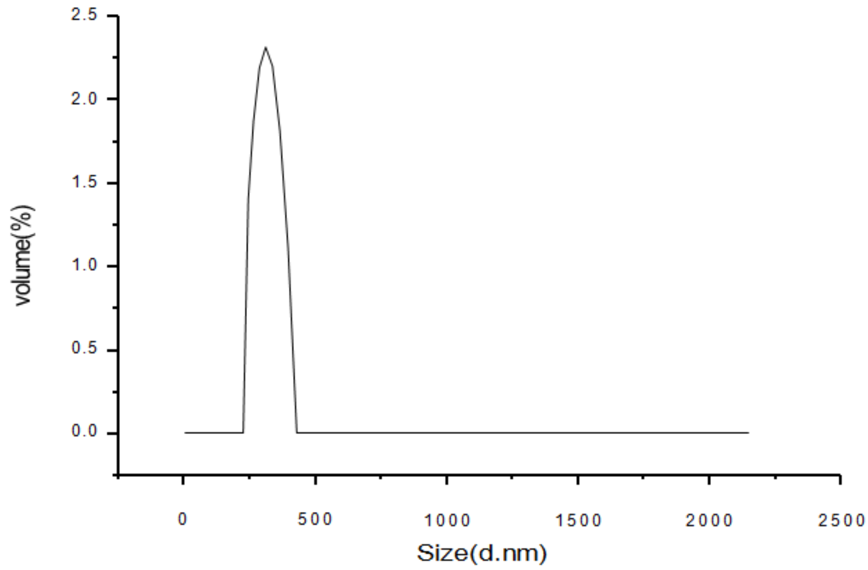
3. Result and Discussion

3.1. Analysis of Particle Size Distribution

By using DLS particle size of iron oxide nanoparticles is measured. Figure 2(A and B) showed below demonstrates it. In figure 2(A), graph showed three peaks with average diameter 85nm, 120nm and 133 nm. From the average diameters, it is concluded that all the nanoparticles are not of same size. After 15 days again size of nanoparticles was measured and developed a change in the peak as shown in figure 2(B). There was only one peak in graph between the ranges of 240nm to 450nm. All other peaks diminished and it was due to increase in size of nanoparticles due to accumulation of smaller nanoparticles [6].



A



B

Figure 2. *(A) PS analysis of fresh samples, (B) PS analysis after 15 days.*

3.2. UV-Vis Analysis

To investigate optical characteristics of produced micro/nanoparticles it is utilized. It's a process in which atoms or molecules' outer electrons absorb radiant energy and shift to higher energy levels. The optical characteristics of the nanomaterial are expressed as absorbance peaks in UV- visible spectroscopy. A Fe^{3+} ion is reduced to Fe^{0} ions when extract is mixed with iron solution. Color of solution varies when electrons are excited. The hue of the solution was altered by adding solution and extract. It took place at room temperature (101). Figure 3 shows spectra of it. From this, it was noticed that nanoparticles showed absorption between the ranges of 250- 350nm but maximum absorption was at 280 nm in the visible range of wavelengths between 200 and 800 nm. Devatha et al. studied UV spectrum of iron nanoparticles and claimed that the top gained for synthesized iron nanoparticles in the range of 300-500nm [7].

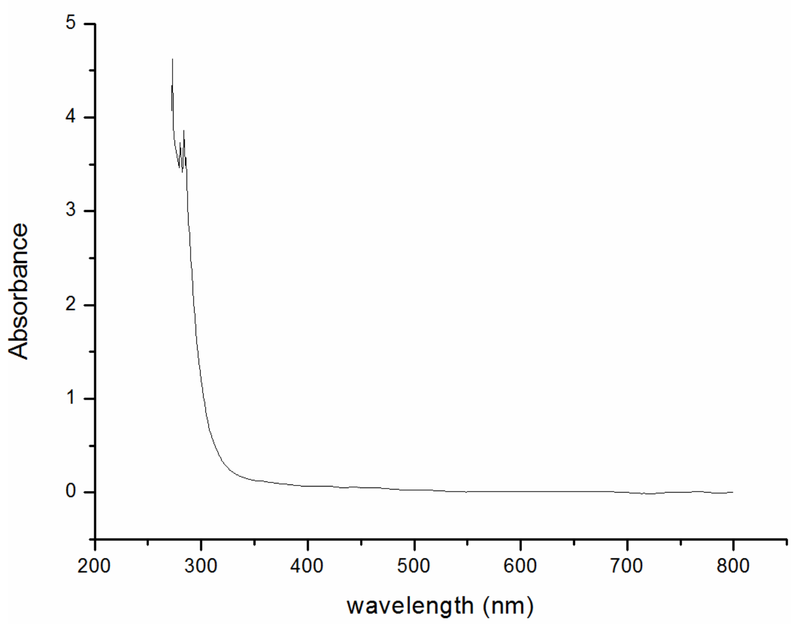


Figure 3. *UV-vis spectra.*

3.3. FTIR of Iron-Oxide Nanoparticles

It showed peak for hydroxyl group at 3190 cm^{-1}. The bands observed on 1610 $cm-^{1}$ for alkene, 1341 $cm-^{1}$ and 1073 $cm-^{1}$ are associated with C-N and ether groups respectively. FTIR spectrum of iron oxide nanoparticles almost matches to the FTIR spectrum of green tea extract. Figure 4 demonstrates the above discussion.

Gottimukkalast al; analyzed the FTIR spectrum of nanoparticles which exhibited peak of hydroxyl group at $3419cm^{-1}$, ethane at $1635cm^{-1}$, $1379cm^{-1}$ for C-N and C-O-C at $1020cm^{-1}$ [8].

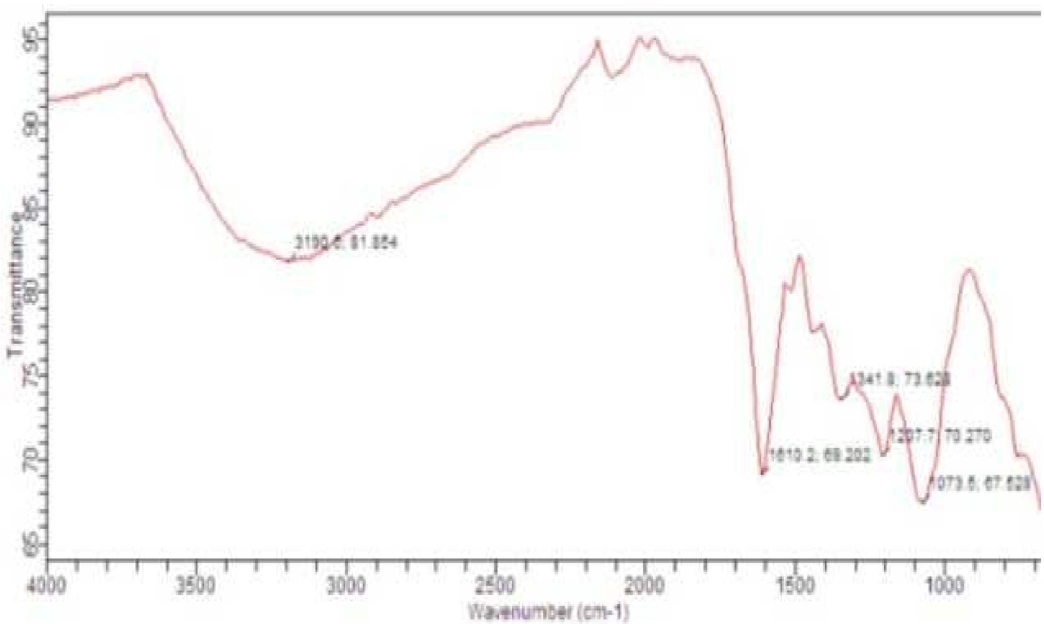


Figure 4. *FTIR of Iron oxide nanoparticles.*

3.4. FTIR of Wet Coating PVP IONPs

The broad band at $3213cm^{-1}$ is due to O-H group. Carbonyl group absorption shifted on 1610 from 1647 became wider because of nanoparticles. Nanoparticles have large surface area so the surface atom coordination is low and this made IR adsorption band wider and FTIR of this showed below in figure 5. This shift in the C=O adsorption band was due to interaction between metal atom and the carbonyl group in PVP. IR adsorptions at $2922cm^{-1}$, $1274cm^{-1}$ and $1073cm^{-1}$ indicate stretching vibrations of C-H, C-N and C-C bond.

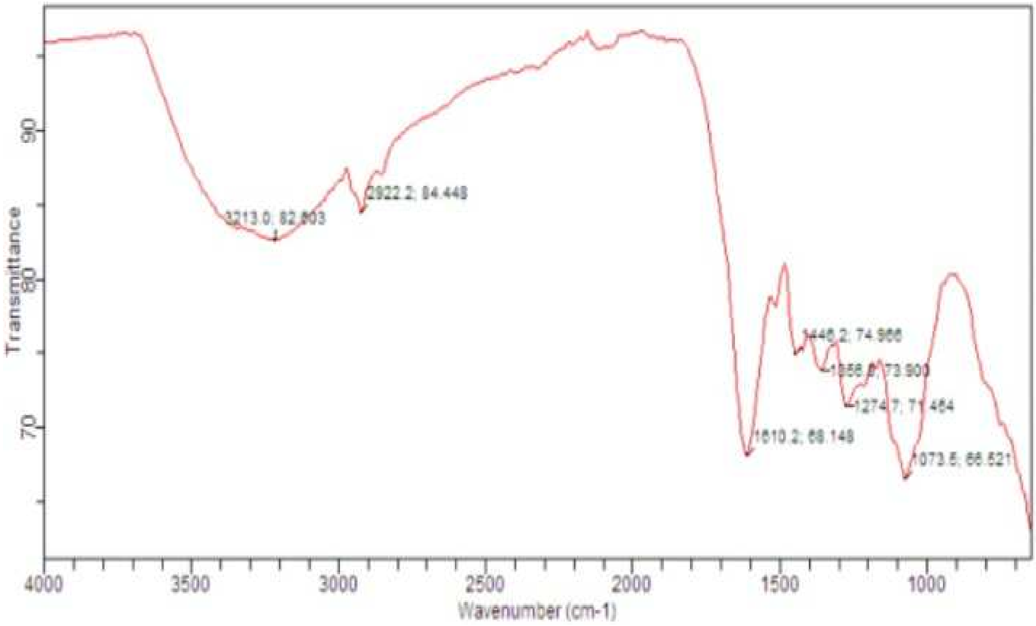


Figure 5. *FTIR of Wet coating pvp IONPs.*

3.5. X-Ray Diffraction Analysis of Iron-Oxide Coated with Polyvinyl Pyrrolidone

The crystal of the sample was determined by XRD. The distinct peaks are found at 2 = 33.17° 35.66°, 38.89°, 49.42°, 54.04°, 57.48° and 62.65° accounting for crystal planes 220, 311, 111, 124, 116, 511 and 440 respectively. All peaks were compared and found to be equal to JCPDS card (19-0629). Figure 6 show the XRD spectrum and illustrate the peaks discussed in above discussion. When PVP was added in it then peaks become broader. This leads to reduction in crystallinity of nanoparticles [9, 10]. The average crystal size is 28.69nm calculated by Scherrer's equation. Crystals have faced centered cubic (fcc) structure.

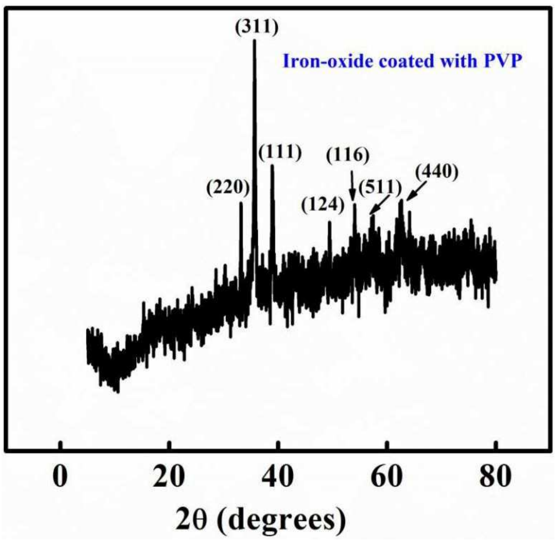


Figure 6. *X-Ray Diffraction Analysis of Iron-oxide Coated with Polyvinyl Pyrrolidone.*

4. Conclusion

From above study it is concluded that nano-particles of iron oxide were formed fortunately from *Camellia sinensis*leaves in less time and easy way. Extract of green tea contains various polyphenols which have the properties of reducing ferric cations and also behave like capping agent. To reduce agglomeration of synthesized iron oxide nanoparticles polyvinyl pyrrolidone, a polymer, is added. UV-visible spectrometer shows highest absorption of iron oxide nanoparticle at 280nm. FTIR disclose about various functional groups. Morphology of them is determined by SEM which shows them spherical but some time they may be heterogeneous. X-ray diffraction analysis shows the crystallinity and size of crystal. Polyvinyl pyrrolidone coated iron oxide nanoparticles have face centered cubic structure.

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