Nigerian Research Journal of Chemical Sciences (ISSN: 2682-6054)
Vol. 7, Issue 2, 2019

SYNTHESIS AND CHARACTERIZATION OF SILVER NANOPARTICLES FROM ORANGE PEELS

*Mustapha, S.; Dallatu, A. Yakubu; Ekwumemgbo, A. Patricia
Department of Chemistry, Ahmadu Bello University, Zaria, Kaduna State, Nigeria.
*Corresponding Author: shehumustapha65@gmail.com

ABSTRACT

In this research green synthesis was done by synthesizing silver nanoparticles from orange peels. Cleaned orange peels were cut into pieces for easy drying. The dried peels were crushed, washed with de-ionised water, air dried and the biomass was boiled for 15 mins, then decanted and filtered. Aqueous solution of silver nitrate was taken to Erlenmeyer flask and different amount of orange peels were added until the colour turned finally to dark brown. The solution was stirred for 35 mins to form silver nanoparticles. Fourier transform infrared (FTIR) spectrophotometer was used to identify functional groups of the orange peels and the synthesized silver nanoparticles which showed the presence of hydroxyl, alkanes, carboxylic, esters and alkanes. UV-Visible spectroscopy was used to quantify the light that was observed and scattered by the sample when the orange peel was at 378 nm and silver nanoparticles was at 474 nm. Both silver nitrate and silver nanoparticles were scanned for confirmatory test of silver nanoparticles which showed the average sizes of the particle to be 26.32 μm for 100 μm scales and 33.33 μm for 80 μm scales. XRD analysis was used to examine the crystal structures of the synthesized silver nanoparticles which provided detailed information on the crystallographic structure and physical properties of the synthesized silver nanoparticle of which the pronounced peak was at 38.9^0.

Key words: Characterization, extract, green synthesis, nanoparticles.

INTRODUCTION

Numerous researches have been carried out by different scholars in the area of nanoparticles synthesis, and characterization [1-4]. Research using carbon nanoparticles for waste water treatment. synthesizing nanoparticles or nanomaterials using biological routes such as those involving micro-organisms, plants, and viruses or their by products, such as proteins and lipids were seen to be better than those manufactured with physical and chemical methods [5]. For example using plant extracts reduces the use of expensive chemicals, consumes less energy, and generates less or nonhazardous chemical by products [6]. Plant extracts base synthesis of nanoparticles uses a bottom- up approach in which synthesis occurs with the aid of reducing and stabilizing agents. Three main steps are followed for the synthesis of nanoparticle using biological system. The choice of solvent medium, the choice of reducing agent, and the choice of nontoxic material as a capping agent to stabilize the synthesized nanoparticle. Silver nanoparticles belong to metallic nanoparticles. Silver is a transition metal and used for medicinal system for curing various diseases. Nanoscale silver received considerable attention in various applications due to its unique properties. Application of nanoparticles in various fields depends on the size, shape and characteristics of the nanoparticles.

Nanoparticles are produced by natural phenomena, and many human industrial and domestic endeavors, such as cooking, material fabrication and transportation utilizing internal combustion and jet engines [7]. Nanoparticles can be derived from larger molecules, or synthesized by 'bottom-up' methods [4]. Silver nano-composite was synthesized for treatment of textile dye [8]. Cadmium was removed from aqueous solution by green synthesis zero-valent silver nanoparticles with benjamina leaves extract [9]. Biological methods for the synthesis of nanoparticles are nontoxic, therefore, eco-friendly and more acceptable than other methods [10]

Nanoparticles size can be controlled by altering the pH of the solution media [10]. Synthesis of nanoperticles using plants extract requires temperature less than 100 ºC or ambient temperature. [10]. Biological agents have been found to be much faster at ambient pressure conditions [10].

Characterization of zinc oxide nanoparticles using Fourier transform infrared spectrum where zinc oxide nanoparticles was analyzed in wide range wavelength between 600 cm^{-1} and 400 cm^{-1}[1]. Characterization of zero-valent iron nanoparticle with multiple techniques such as XRD, TEM, FTIR, and UV-Vis spectroscopy, where a broad band peak on XRD reveals existence of amorphous phase of iron [11]. Characterization of metallic nanoparticle where the result showed strong absorption maximum in the range of 500-600 and 400-450 nm, respectively, while using XRD result showed a broadening of the peaks which confirms the formation of particles in nano size [12].

Nanotechnology has been receiving tremendous attention by researchers due to its effectiveness in addressing water problems related quality and quantity. Furthermore, nano materials are contributing to the development of most efficient treatment process among the advanced treatment technology. Hence, the aim of the research is green synthesis to synthesize and characterized silver nanoparticles.

MATERIALS AND METHODS

Plants used for this nanoparticles synthesis were orange peels obtained from orange fruits. The equipment used where Digital weighing balance, pestle and mortar, filter paper, magnetic stirrer, desiccators, beakers, conical flask, sample bottles, volumetric flask, measuring cylinder, funnel, spatula, reagents bottles, UV-visible spectrophotometer, scanning electron microscopy and FTIR spectrophotometer.

Preparation of Orange Peels

The method as reported by Aina, was adopted in extracting peels from the orange fruits. Fresh orange fruits obtained from Samaru market, in Zaria, Kaduna State, Nigeria, were examined for its suitability and usability. Each of the fruits was washed several times using tap water, then with de-ionized water to remove impurities. The orange peels were cut into smaller pieces for easy drying. The dried peels were crushed, washed with de-ionised water and air dried. The biomass was boiled for 15 mins before being decanted and filtered using filtered paper [13].

Synthesis of Silver Nanoparticles

The method as reported by Priya was adopted. Aqueous solution of silver nitrate (10 mL of 10 mM) was taken to Erlenmeyer flask. Different amounts of orange peels were added (up to-30 mL) until the colour turned finally to dark brown. The solution was stirred for 35 mins to form silver nanoparticles. The colour changed to brown within 25 min, showing that silver nanoparticle was formed [8].

Characterization of the Synthesized Nanoparticles

Characterization of Nanoparticles is significant to evaluate the nature of the nanoparticles.

FTIR Spectrophotometric Analysis

FTIR spectrophotometer (630 Agilent Technologies) was used to identify some functional groups of the orange peels and the silver nanoparticles synthesized.

UV-Visible Spectroscopy

UV-Visible spectroscopy was used to quantify the light that was observed and scattered by the sample. Both silver nitrate and silver nanoparticles were scanned for confirmatory test of silver nanoparticles.

Scanning Electron Microscope (SEM) Analysis

The morphology of the synthesized silver nanoparticles was recorded using a scan electronic microscopy (phenom prox, MVEO16477836).

X-ray Diffraction (XRD) Analysis

This was used to examine the crystal structures of the synthesized silver nanoparticles. It is a method of determining the arrangement of atoms within a crystal, in which a beam, X-ray, strikes a crystal and diffracts into many specific directions using a diffractometer. It provides detailed information on the crystallographic structure and physical properties of materials and thin films

RESULTS AND DISCUSSION

The biogenic synthesis of silver nanoparticles from orange peel extract was carried out. Silver nitrate used has distinctive properties. As the orange peel extract was mixed with the aqueous solution of silver nitrate, there was change of colour from pale brown to dark brown due to the reduction of silver ion. The formation of silver nanoparticles was successful as suggested by initial changes in colour. Silver nanoparticles exhibit brown colour in aqueous solution due to excitation of surface plasmon vibration in silver nanoparticles. The result agrees with the finding of different researchers [6, 14].

FTIR Spectrophotometric Analysis

The FTIR of orange peel showed weak broad peak at 3212.6 cm^{-1} corresponding to O-H stretching vibration [15] of carboxylic. The bands at 2944.6 cm^{-1} and 2840.1 cm^{-1} with weak peaks correspond to the C-H stretching of alkane CH_3, CH_2 Or CH [16]. The region around 1733.2 cm^{-1} and 1802.8 cm^{-1} is attributed to absorption by esterified and free carboxyl groups [16] while 1520 cm^{-1}, 1416 cm^{-1},1360 cm^{-1} and 1274.7 cm^{-1} are due to asymetric stretching and

C-H rocking of alkane. Strong broad peak at 1010.1 cm^{-1} corresponds to C-O stretching vibration of ester [16].

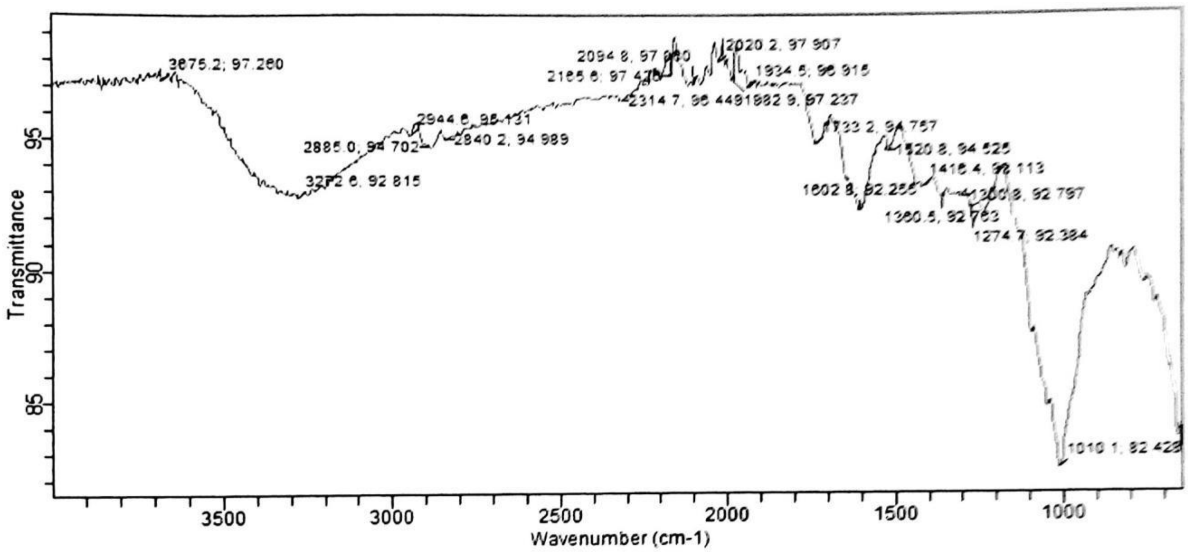


Figure 1: FTIR of Orange Peels

Silver Nanoparticles

The FTIR of silver nanoparticles with strong broad peak at 3276.3 cm^{-1} corresponds to the O-H stretching vibration [15] of carboxylic. The medium peaks at 2922.2 cm^{-1} and 2855.1 cm^{-1} correspond to C-H stretching of methylene [16], while 1457.4 cm^{-1}, 1375.4 cm^{-1} and 1274.7 cm^{-1} are due to symetric stretching. There is a strong broad sharp absorption at 1021.3 cm^{-1} which corresponds to C-O stretching vibration of ester [16].

However, as the result of characterizing silver nanoparticles and orange peels, some changes were observed in the spectra. The weak broad peak which occured at 3212.6 cm-1 in the orange peel spectra changed to strong broad peak which occured at 3276.3 cm^{-1} (O-H Stretching of carboxylic group) in silver nanoparticles. 2944.6 cm^{-1} and 2840.1 cm^{-1} were shifted to 2922.2 cm^{-1} and 2855.1 cm^{-1} (C-H Stretching) , also 1733.2 cm^{-1} and 1802.8 cm^{-1} were also shifted to 1808.5 cm^{-1} which is attributed to absorption by esterefied and free carboxylic groups and 1010.1 cm^{-1} shifted to 1021.3 cm^{-1} C-O Stretching vibration of ester group).

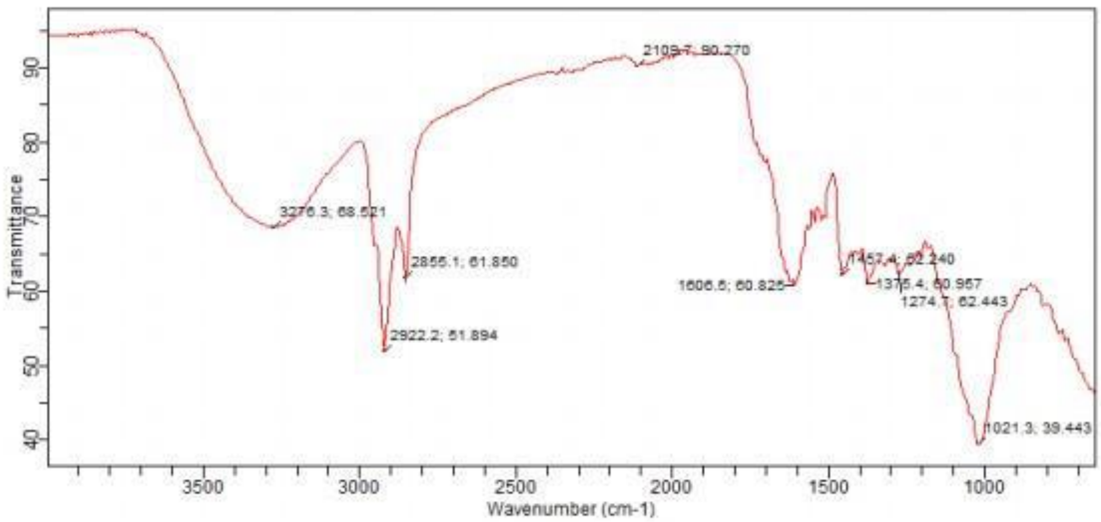


Figure 2: FTIR of silver nanoparticles

UV-Vis Spectroscopy

The UV-Vis spectrum of orange peel extract solution has absorbance peak at 378 nm, which agrees with the result of Al;-qahtani [14].

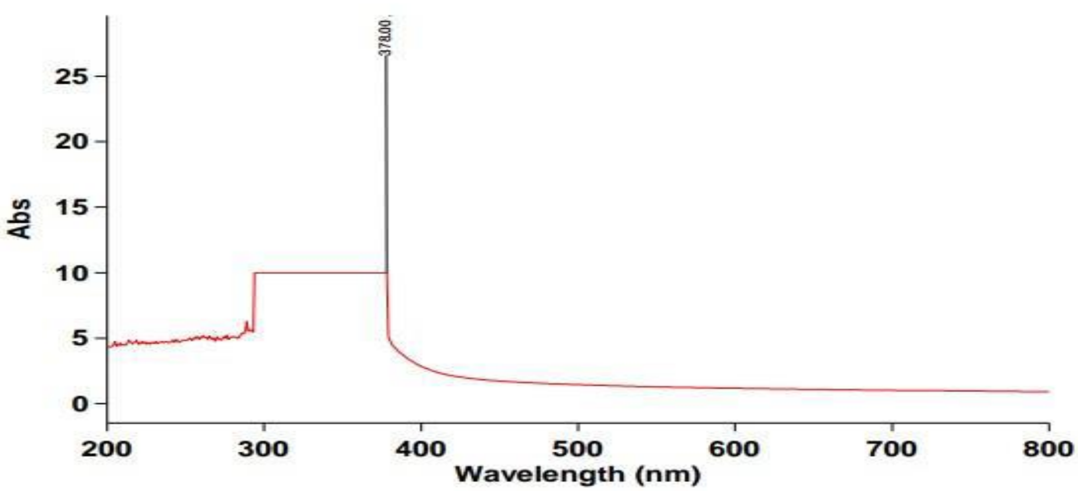


Figure 3: UV-Vis spectroscopy of orange peel extract solution

UV-Vis spectroscopy of silver nitrate

The UV-Vis spectroscopy of silver nitrate solution has absorbance peak at 266 nm.

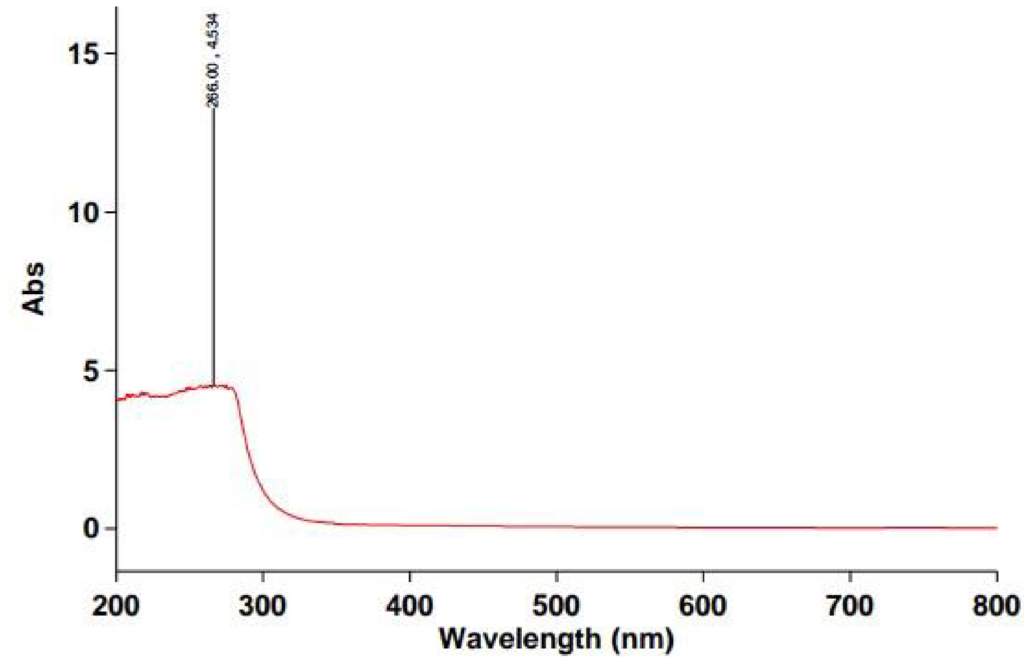


Figure 4: UV-Vis Spectroscopy of Silver Nitrate

UV-Vis Spectroscopy of Silver Nanoparticles

The UV-Vis spectroscopy of silver nanoparticles with absorbance peak at 474 nm indicates the formation of silver nanoparticles. UV-Vis spectroscopy was used to confirm the presence of nanoparticles. This was consistent with the finding of Al;-qahtani [14] in which all the absorbance peak were reported to be within 400-500 nm .

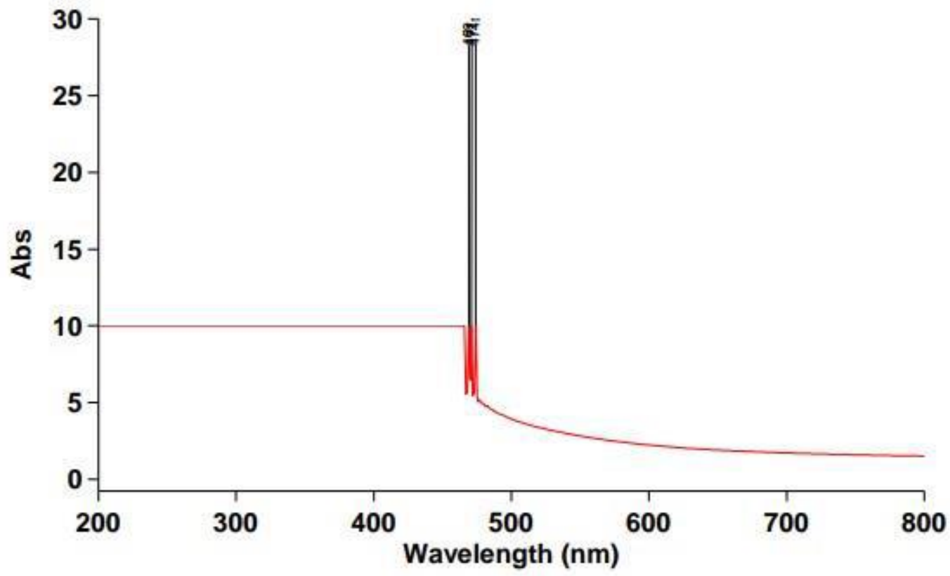
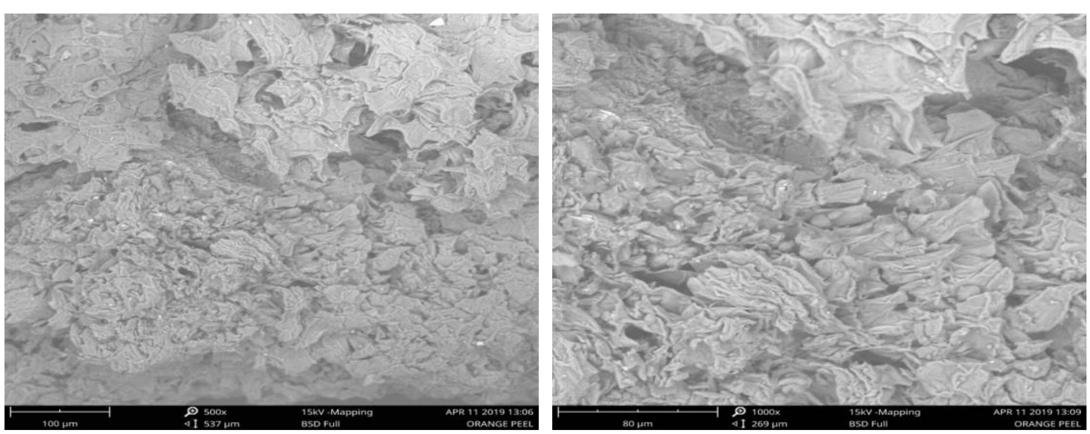


Figure 5: UV-Vis Spectroscopy of Silver Nanoparticles

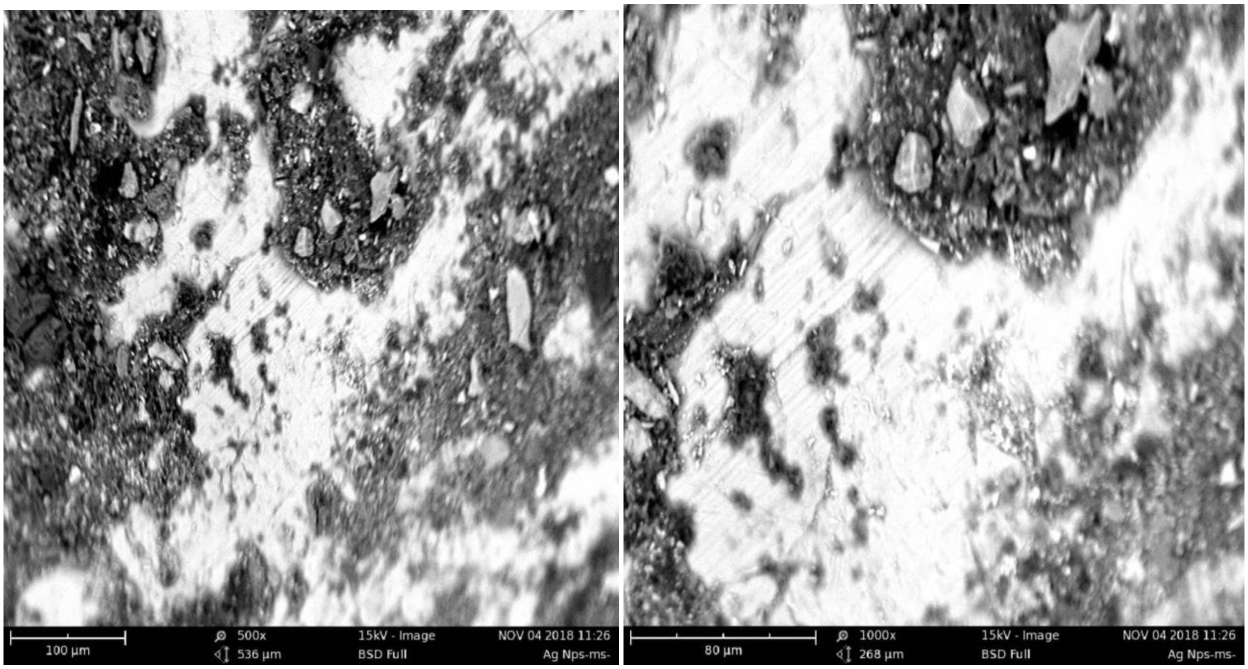
SEM Image Analysis

The SEM was employed to analyze the structure and morphology of the nanoparticles to give further insight into the features of the silver nanoparticles and orange peels obtained.



SEM Image of Orange Peel at 100 µm *SEM Image of orange Peels at 80 µm*

Figure 6: *SEM Image of Orange Peels at 100 µm and 80 µm*



SEM Image of silver nanoparticles at 100 µm *SEM Image of silver nanoparticles at 80 µm*

Figure 7: SEM Image of silver nanoparticles at 100 µm SEM Image of silver nanoparticles at 80 µm

SEM images of both orange peel and silver nanoparticles were employed to analyze the structure and morphology of the nanoparticles to give further insight into the features of the silver nanoparticles and orange peels obtained. The average sizes of the particle were 26.32 µm (for 100 µm scale) and 33.33 µm (for 80 µm scale) as shown in Figures 6 and 7

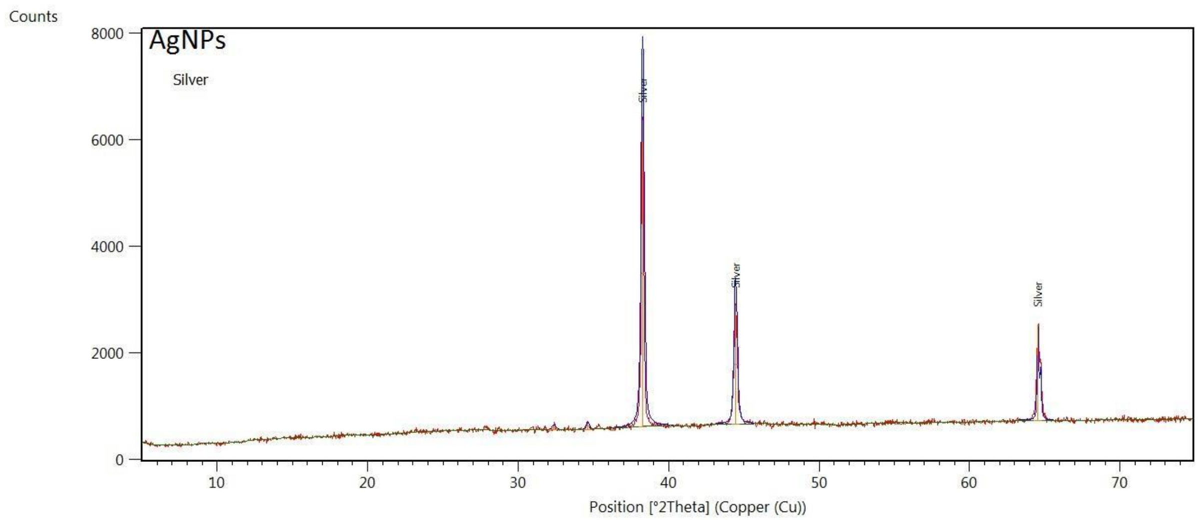


Figure 8: XRD pattern of Silver nanoparticles

XRD is a powerful technique used to identify the crystalline phases present in materials and to measure the phase composition, preferred orientation, grain size, strain state and defect structure of these phases. It is a method of determining the arrangement of atoms within a crystal, in which a beam X-ray strikes a crystal and diffracts into many specific directions using diffractometer. It provides detailed information on the crystallographic structure and physical properties of materials and thin films. XRD result showed a broadening of the peaks which confirms the formation of particles in nano size.

CONCLUSION

The synthesis of silver nanoparticles from orange peels extract was carried out. The silver nanoparticles exhibit brown colour in aqeous solution due to excitation of surface plasmon vibrations in silver nanoparticles. The synthesis of silver nanoparticles was confirmed by measuring the UV-Vis spectrum which showed absorbance peak at 466 nm. The FTIR analysis showed the functional groups present in orange peels and silver nanoparticles like the presence of –OH, -CH, -CO-. SEM was used to analyze the structure and morphology of the nanoparticles to give insight into the features of the silver nanoparticles as pore size aids adsorption, while XRD was used to determine its crystalinity. XRD result showed a broadening of the peaks which confirms the formation of particles in nano size.

REFERENCES

1. Nadia, M.A. & Yousef, N. S. (2015). Synthesis and characterization of zinc oxide Nanoperticle for the removal of **Cr** (vi*). International jounal of scientific and engineering research*, 6(7), 1-7.
2. Santosh, K.K. & Sharada, S. (2015). Development, synthesis of zinc oxide nanoparticles and its application in water treatment technology. *Journal of technical research india 2(12).*
3. Sezging, N., Sahim, M., Yalcin, A. & Koscogcu, Y., (2015). Synthesis, characterization and the heavy metal removal efficiency of Mfe2O4 (M=Ni, Cu) nanoparticles. *Ekoloji*, 22(89), 89-96
4. Samanta, H. S., Das, R. & Bhattachanjeec. (2016). Influence of nanoparticles for waste water treatment. *Austing Chemical Engineering,* 3(3), 1-6.
5. Khaydarov, R. & Gapurora, O. (2010). Application of carbon nanoparticles for water treatment, NATO science for peace and security series C. *Enviromental Security*, 253-258.
6. Benakashani, F. (2016). Biosynthesis of silver nanoparticles usig Caparris spinosa Leaf extract and thei antibacterial activities. Biosynthesis of silver nanoparticles using Capparis spinosa L. leaf extract and their antibacterial activity, *Karbala International Journal of Modern Science*, http://dx.doi.org/10.1016/j.kijoms.2016.08.004.
7. Scientific Committee on Emerging and Newly Identified Health Risk (2016).
8. Priyanka, K. (2017). Effective adsorption of cadmium (II) ion on orange peels. *International Research Journal of Engineering and Technology*, 4(6), 1407-1409
9. Khairia. M., & Al-qahtani (2017). Cadmium removal from aqueous solution by green synthesis zero- valent silver nanoparticles with benjamina leaves extract. *The Egyptian Journal of Aquatic Research,* 43(4),269-274
10. Jayanta, K.P. & Kwang-Hung, B. (2014). Green Nanobiotechnology: Factors affecting synthesis and characterization techniques. *Journal of Nanomaterials*, vol. 2014, Article ID 417305, 12 pages, https://doi.org/10.1155/2014/417305.
11. Ritu, S., Virendra, M. & Rena, P.S. (2011). Synthesis, characterization, and role of Zero-valen nanoparticle in removal of hexavalent chromium from chromium spiked soil. *Journal* of *Nanoparticle Research*, 13(9), 4063-4073.

12. Manish, S.R., Ashok, K.S., Sarita, S., & Sanjay, V. (2015). Removal of lead (II) from aqeous solution by orange peels. *International Journal of Applied Research*, 1(9), 911-413.

13. Aina, V.O., Barau, M.M., Mamman, O.A., Zakari, A., Hanma, H., Umar, M.S..H. & Abba, Y.B. (2012). Extraction and charactering pectin from peels of lemon, grape, fruit and sweet orange. *British Journal of Pharmacological Toxicology*. 3(6), 259-262

14. Al;-qahtani, K.M. (2017). Cadmium removal from aqueous solution by green synthesis of zero-valent silver nanoparticle with benjamina leaves extract. *Egyptian Journal of Aquatic Research*

15. Coates, J. (2000). Interpretation of infra red spectra, a practical approach Encyclopedia of Analytical chemistry, R.A. Meyers (ed). O John Wiley and son Ltd Chichester, 10815-10837

16. Chandra Sekhar, K., Kamala, C.T., Chary, N.S. & Anjaneyulu, Y. (2002). Removal of heavy metals using a plant biomass with reference to environmental control. *International Journal of Mineral Processing*, 68, 37-45.