

Eco-Friendly Method for Green Recovery of Silver Nano Particles from Effluent Fixer Solution through interacting with *Q. brantii* (Oak) Peel Hydro Alcoholic Extract

Sedigheh Mirzaei, Hamid Reza Safaei*

Department of applied Chemistry, Shiraz Branch, Islamic Azad University, PO Box 71993-5, Shiraz, Iran

*Corresponding author: Hamid Reza Safaei. Tel.: +98(71)36402715; E-mail: harsafaei@yahoo.com; safaei@iaushiraz.ac.ir

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ABSTRACT

Silver nano particles (AgNPs) are gaining in popularity for their strong antimicrobial activity, low toxicity towards humans. Despite various methods are used for recovery of metallic silver from its waste solutions, none of them consider the synthesis of silver nano particles. Biological synthesis of nanoparticles is a relatively new emerging field of nanotechnology. Different biological methods are gaining recognition for producing of silver nanoparticles (Ag-NPs), but in all of these methods, standard solution of AgNO_3 is used as precursor of silver. In this study fixer effluent solution as primary source of silver and *Q. brantii* (Oak) peel hydro alcoholic extract as a bio-reducing and bio-capping agent were used. In this method no need to chemical and in compliance with principles of green chemistry. The best conditions of reaction such as mixing ratio of waste to Oak extract, pH, temperature, time period and concentration were investigated by evaluation obtained Ag NPs by UV-Visible and FT-IR spectroscopy, DLS technique, SEM and XRD.

Keywords: *bio-recovery, silver nanoparticle, waste photography solution, green synthesis, (Oak) peel hydro alcoholic extract*

INTRODUCTION

The increase in industrial activities has intensified to demand for more raw materials and despite the fact that, for some of them special precious metals availability has become more and more limited [1]. Technology growth has received significantly more attention globally over the last few years. Its growth has been very visible through the consumption of resources. The resources required to sustain the current level of economic growth may not be available over the next decades. Therefore, beside the other factors, Constraints on available resource may limit global economic growth over the upcoming decades. This is the vision of the future emerging from a major report launched by the Actuarial Profession in association with Anglia Ruskin University on 17 January 2013. It also suggests that financial models fail to factor in the risks of climate change and resource scarcity. Recent studies have shown that mineral resource scarcity is significant as well as the much discussed issue of energy scarcity [2]. These findings point firmly in the direction of recycling, as limited resources can be used preferentially to recycle materials in order to sustain mineral resources. This mineral scarcity has the potential to make recycling a necessity. Tons of wastewater containing heavy metals are generated every year [3] and with time, waste is increasingly seen as a valuable resource for industry. Recycling changes not only the scale and location of production, but also value centers in the production consumption chain. Moreover, Metals are among the most commonly encountered and difficult to treat environmental pollutants. The combination of rapidly expanding industry and increases in domestic activities has caused significant environmental problems due to metal ion pollution and critical losses of non-renewable metal resources [4]. Considering above mentioned, Recovery of metals from secondary sources is a necessary undertaking in the twenty-first century, due to protection of the environment and conservation of natural reserves of the elements.

In the last decades, there has been a considerable expansion in the use of precious metals in medicine, optical devices, electronics and catalysis [5]. The wastewater of these industries contains high concentrations of precious metal such as silver. The waste effluent solution of X-ray photographic films

containing metallic silver are a good source for silver recovery [6]. It has been reported that 25% of the world's silver needs are provided by recycling and more than 70% of this, is obtained from photographic fixer solution [7]. The fixer solution is a photographic waste solution of ammonium or sodium thiosulphate that applied for conversion of the silver chloride or bromide into water-soluble silver thiosulphate. Then, this water-soluble silver salt transfer from the film into the fixing bath [8]. Another photographic waste solution that contains silver is the spent stabilizer solution. Wastewaters containing free silver ion is very toxic to aquatic organism even at trace concentrations [9,10]. Hence, the removal of silver from these wastes solution is of both economic and environmental benefit. Despite widespread methods are used for recovery of metallic silver from its waste solutions such as Electrolysis, metallic replacement, ion-exchange, chemical precipitation, reverse osmosis and membrane processes, none of them consider the synthesis of nano particle size of silver.

In recent years, Silver nano particles (AgNPs) are popular for their strong antimicrobial activity [11-14] and its relatively low toxicity towards humans [15]. Besides their antimicrobial features, silver nanoparticles also exhibit strong optical features making them suitable for biological sensing and imaging [16]. Silver nanoparticles are also applied in conductive inks, adhesives and pastes for electronic devices, due to their excellent conductivity [17]. They are also used as catalysts in several chemical reactions [18, 19]. Two major routes can prepare silver nanoparticles; the first one is a physical approach such as evaporation/condensation and laser ablation. The second one is a chemical approach in which the metal ions in solution is reduced by chemicals included borohydride, or elemental hydrogen [20-26]. In chemical reduction the reduction of Ag^+ ions lead to the formation of silver atoms (Ag°), as colloidal dispersions in solvents. It is important to use protective agents or capping agent to stabilize dispersive nanoparticles during the course of silver nanoparticle preparation. The most common strategy is to protect the nanoparticles with protective agents that can be absorbed on or bind onto the nanoparticle surface, avoiding their agglomeration [21, 27].

Plant extracts are often environmentally and economically

friendly materials. In the last decade, the use of live plants or whole plant extract and plant tissue for reducing silver ions to nanoparticles has attracted considerable attention [28-30]. All parts of a plant including leaves, fruits, roots, seeds, bark and stems, can be used in the synthesis of silver nanoparticles (Ag NPs), replacing potentially hazardous chemicals. The reason plant extracts work so well in the synthesis of nanoparticles is because they act as reducing agents as well as capping agents [29-31]. Although there are several synthetic routes to make silver nanoparticles as mentioned above, however, none of them has noted to do recovery as a goal. They have used standard solution of silver nitrate as a source of silver ions, while there is substantial difference between the chemical composition of photography waste solution and standard solution of AgNO_3 [32]. Consequently, there is a need for method of silver nanoparticles recovery from photographic waste solution that is efficient, selective, and use environmentally benign materials or processes. A number of few research group were used a various parts of Oak plant for chemical examinations. Recently, Korbekandi and his group have used oak leaves hydro alcoholic extract for synthesis spherical, and poly-dispersed nano size of silver from standard solution of silver nitrate [33]. Later, Heydari and Rashidipour have reported Ag^+ in standard solution of AgNO_3 can be reduced to Ag NPs by interacting with oak Fruit Hull aqueous extract in the present of ammonia solution. In this effort, pH of the solution was adjusted to the desired value by using sodium hydroxide or phosphoric acid solution and the Ag NPs were obtained with 40 nm average particle size [34].

In continuing, our general interest to synthesis of biological active compounds and green recovery system [35-41]. We herein report an efficient bio-recovery of silver nanoparticles from photography waste solution by environmental benign process. In the present study, oak peel was used as a bio-reductant for the recovery of silver nanoparticles from photography waste solution. To the best of our knowledge, no such process for recovery of silver nanoparticles from silver waste solution has been reported to date. The reason behind selecting this particular plant is Persian oak, *Quercus brantii* Lindl., is one of the most important and abundant plant species in Iran specifically in Zagros Mountains zone. Furthermore, extract of *Quercus infectoria* was found to contain a large amount of polyphenols and flavonoids and possess a potential reducing power [33, 34]. Moreover, it is commercially economic and abundantly available. Oak Peel traditionally employed as an anti-diarrheal [42]. In medicine, it has several properties, such as albumin precipitate and is to create a protective coating for tissue [43]. Furthermore, oak fruit infusion for the treatment of diseases like Diarrhea, stomach ache, Hemorrhoid, Anemia, Burn, rickets and strengthen the body used [44]. Due to the numerous applications of oak, thereby has promising availability for the commercial recovery of nano-scale silver.

2. MATERIALS AND METHODS

2.1. Materials

Fresh *Q. brantii* was collected from local source, Iran, Kazeron. Reagents were purchased from Merck, Fluka or Aldrich and they were used as received without further purification. The industrial effluent used in the recovery process of silver nano-particles were fixing bath from the Shiraz central hospital (MRI) and Shiraz university of medical science photographic waste solution. Silver concentration of these baths were 8 gL^{-1} .

2.2. Experimental procedure

2.2.1. Preparation of extract

Peel of *Q. brantii* (oak) was isolated and were washed several times with water to remove the dust particles then were dried at 25°C in shadow. The dried peel of *Q. brantii* were cut into small pieces. In order to prepare hydro-ethanolic extract of *Q. brantii*, 10 g of dried prepared peel of *Q. brantii* was extracted for 20 min with 100mL ethanol 50% and filtered by using Whatman filter paper. Finally, the filtrate was centrifuged for 10min at 4000 rpm. This extract was stored at 4°C for further experiments.

2.2.2. Determination of silver concentration in photography waste solution

The concentration of silver in waste solution of silver ion with thiosulfate as counter ion was measured via the method that have been reported by Murphy [45]. For this measurement fixer effluent solution (25 mL) was transferred to a 250 mL beaker. Then distilled water (100 mL), sodium acetate (20 mL; 50 g L^{-1}) and EDTA (20 mL; 50 g L^{-1}) were added to the beaker. Adjust the pH of the solution between 6-7 using ammonia hydroxide and acetic acid. Afterwards, hexamminecobalt (III) chloride solution (30 mL ; 30 gL^{-1}) was added. The color of the solution was changed to amethystine. Then, stirred the suspension solution until a quantitative precipitate was formed. Then the suspension solution was filtrated, washed with distilled water and dried at 95°C until a constant weight was obtained. The conversion factor to silver is 0.2187.

2.2.3. Recovery of silver nanoparticles

In order to determine the optimum parameters of the bio-recovery process, the Factorial Design of Experiments were that the one factor was varied at a time keeping the other factors constant. concentrations of fixer effluent solution (8 gL^{-1}) was interacted with the prepared peel oak extract in two different mixing ratios of fixer effluent and peel oak extract (1:30, 1:40) in 70°C within 15 min time period with vigorous stirring. Moreover, the size, shape and dispersity of Ag NPs were investigated under various temperature, time of reaction and pHs by adjusting with 0.5N NaOH. The formation of nanoparticles if any were used for the subsequent characterization studies. All experiments performed in this work were repeated three times. The result reported represents the average of the values obtained for each reaction.

2.3. Characterization

The silver nanoparticles were preliminary characterized by UV-Visible spectroscopy. The spectra were recorded on HACH UV-Vis Spectrophotometer DR 5000 single beam spectrophotometer from 200 to 700 nm after dilution with distilled water. The solids of suspension were then subjected to FTIR spectroscopy measurements. The measurements were carried out on a PerkinElmer Spectrum RXI. The particle size distribution of the nanoparticles was determined using dynamic light scattering (DLS) HORIBA Light Scattering Particle Size Analyzer LB-550. Particle size along with its poly-dispersity were assessment based on measuring the time dependent fluctuation of laser light scattering undergoing Brownian motion. Scanning Electron Microscope of nanoparticles were performed by KYKY-EM3200 Digital Scanning Electron Microscope. SEM images were carried out by drop coating the dispersion containing the particles onto a glass slide after required reaction time. Afterwards, the obtained coating was scanned at a rate of 100 mV s^{-1} in the range $50\mu\text{m}\times 50\mu\text{m}$. The obtained nano-powders was subjected to X-ray diffraction analysis, Philips PW-180, Netherlands. The target was Cu K_α with wavelength of 1.5406 \AA and the scanning range was selected between 10° and 100° under the 40 kV generator power and 30mA current.

3. RESULTS AND DISCUSSION

3.1. Optimization of conditions

3.1.1. Optimization of mixing ratio

Preliminary, the formation of silver nanoparticles was monitored through visual assessment. The reaction mixture turned to brown color from red brown color within 15 min indicated the synthesis of silver nanoparticles (Fig. 1).



Fig 1. bio-recovery of Ag-NPs from fixer effluent solution of Ag^+ interacted with the prepared peel Oak extract

For obtaining the best conditions the fixer effluent solution (Hospital; 8 gL^{-1} , 30mL) was heated to 70°C . Then, 1mL of prepared peel oak extract was interacted with 30 mL and 40mL fixer effluent solution, separately. Afterwards, the temperature was kept at 70°C within 15 min time periods with vigorous stirring. Determination of particle size of obtained metallic silver could be the key factor for efficiency evaluation of each experiment. Particle size of obtained Ag-NPs were determined by measuring the random changes in the intensity of light scattered from a suspension or solution of them (Fig. 2). This technique is commonly known as dynamic light scattering (DLS). The particle size determined by dynamic light scattering is the hydrodynamic size. That is, the determined particle size is the size of a sphere that diffuses the same way as target particle. (Table 1, Fig. 2)

Table 1 mean value of Ag particles size in two different mixing ratios of fixer effluent solution and peel Oak extract

| | a | b |
|---|------|------|
| mixing ratios fixer effluent : peel Oak extract | 30:1 | 40:1 |
| Mean value of Ag particle size | 62 | 91 |

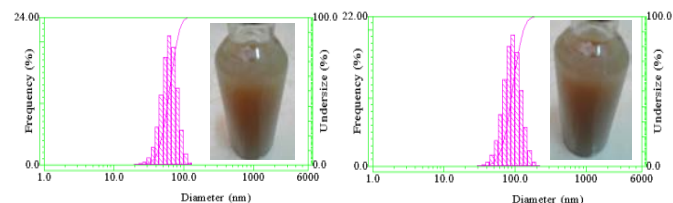


Fig 2. DLS results for interaction of different mixing volume ratios of fixer effluent: peel Oak extract at 70°C within 15 min time period a) 30:1, b) 40:1

We found that particles size of metallic Ag was obtained 62 nm when 1mL of peel Oak extract was interacted with 30mL of fixer effluent (Fig. 2-a). This particles size increased by raising the amount of fixer effluent solution up to 40 mL (Fig. 2-b). Moreover, Fig 2-a shows the distribution of particle size is narrow. It is worth to mention that homogeneity of these particles is important in many applications [46].

3.1.2. Optimization of pH, temperature and time period of reaction

As mentioned above the formation of silver nanoparticles was accomplished with browning the color of mixing solution (Fig.

1). So, UV-Visible spectroscopy is a good, cheap and reliable method for evaluation of formation nanoparticles of silver.

Light wavelengths in the 300-800 nm are generally used for characterizing various metal nanoparticles [47]. Spectrophotometric absorption measurements in the wavelength ranges of 400-500 nm [48] are used in characterizing the silver nanoparticles. This characteristic peak is due to the oscillation of conduction band electrons of Ag-NPs known as the surface plasmon resonance (SPR) [49]. UV-vis spectroscopy was ascertained to check the formation of Ag-NPs at pH 6.

For assessment of the formation of Ag-NPs in the upper pH near to neutral condition, the reaction was done under optimized ratio of fixer effluent solution to peel Oak extract (30:1) at pH 6 by addition of NaOH (0.7mL, 0.5N) to pre heated fixer solution at mentioned temperature(70°C) for 5 min time period with stirring. Then, the reaction was followed with the addition of peel Oak extract (1mL) and stirring the mixture for additional 15 min. During this time the color of the mixture was changed to brown. The UV-VIS spectrum of result solution is shown in Fig 3.

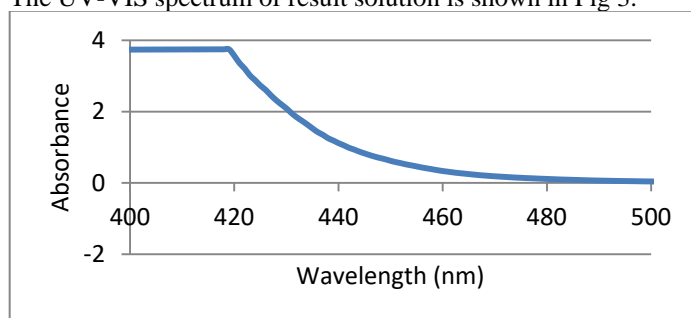


Fig 3. UV-VIS spectrum correspond to interaction of effluent fixer solution and peel Oak extract under optimized ratio at pH 6. The UV-VIS spectrum was shown one absorption peak arising at 418 nm. It means that the Ag-NPs was formed at pH 6. In fact, a major influence of pH is its ability to change the electrical charges of biomolecules which might affect their capping and stabilizing abilities and subsequently the growth of the nanoparticles [50].

In another try to evaluate feasibility of the reaction in milder condition we did the reaction at 35°C . The color was changed to brown after 48h. Then after for assessment of resulted mixture, UV-vis spectra were taken. (Fig 4)

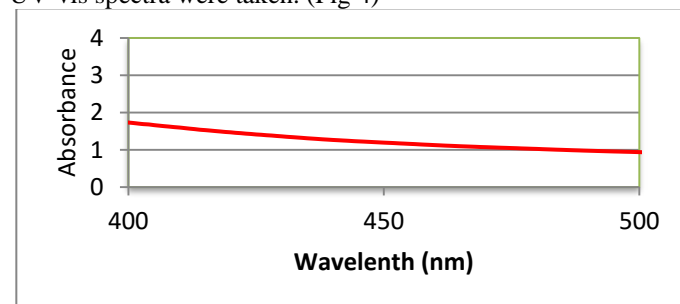


Fig 4. UV-VIS spectrum of resulted mixing of effluent fixer solution and peel Oak extract under optimized ratio at 35°C . Unfortunately, the UV-VIS spectrum was shown there is no absorption between 400-500 nm. So, in contrast to other silver nanoparticles made methods from nitrate solution of Ag^+ [50], at this temperature Ag-NPs was not formed. Thus, 70°C was chosen for our bio-recovery of Ag-NPs as optimal temperature.

In order to determination of minimum time of interaction for obtaining best results the reaction was carried out in 15 and 30 min under optimized conditions by UV-VIS spectroscopy. The UV-VIS spectrum of resulted solutions are shown in Fig 5. With

reviewing of Fig. 5 it can be understanding that Ag^+ completely reduced to form silver nanoparticles within 15 min. It can be confirmed by the fact that there is no further change of UV-VIS spectra after elongation time of interaction solution.

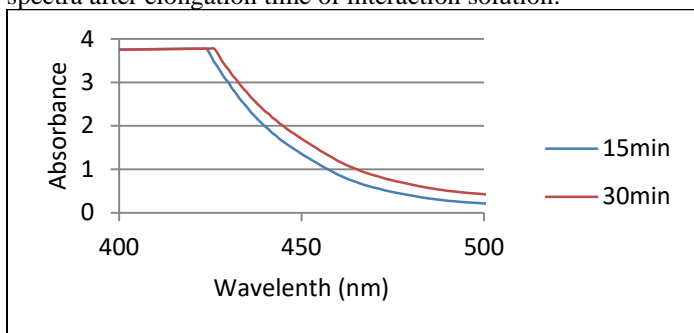


Fig 5. UV-VIS spectrum of resulted mixing of effluent fixer solution and peel Oak extract under optimized conditions in various time period

3.2. Stability of synthesized Ag-NPs

In order to determine the stability of green recovered of silver in the form of Ag-NPs, after interaction of effluent fixer solution and peel Oak extract under optimized condition, the obtained solution of nanoparticles was stored at 4 °C under air condition for 30 days. After this time, without any shaking or stirring, durability of nanoparticle solution was evaluated by UV-Visible spectroscopy (Fig 6).

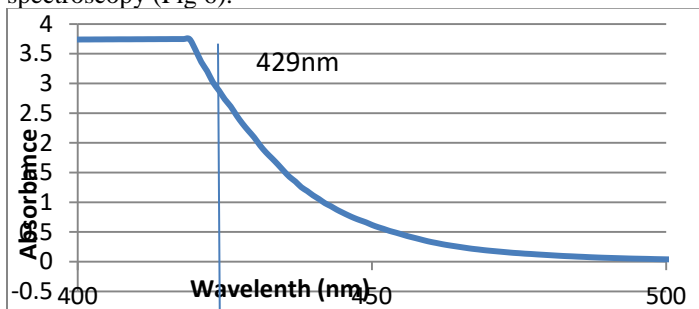


Fig 6. UV-VIS spectrum of resulted mixing of effluent fixer solution and peel Oak extract under optimized conditions after 30 days

Absorption peak at 429 nm in UV-Visible spectrum of obtained mixing solution was confirmed the stability of nanoparticles even after 30 days, which can be attributed to surrounding of nanoparticles by organic constituent, that born with peel Oak extract.

3.3. Characterization of Ag NPs

3.3.1. UV-Visible spectra

As mentioned above UV-Visible spectroscopy is used for monitoring the signature of silver nanoparticles due to strong surface plasmon resonance absorption in the visible region. The UV-VIS spectrum of peel Oak extract before and after interacting with waste photography solution at optimized conditions was shown in fig 7. Fortunately, there is no interference absorption between peel Oak extract and Ag NPs spectrum.

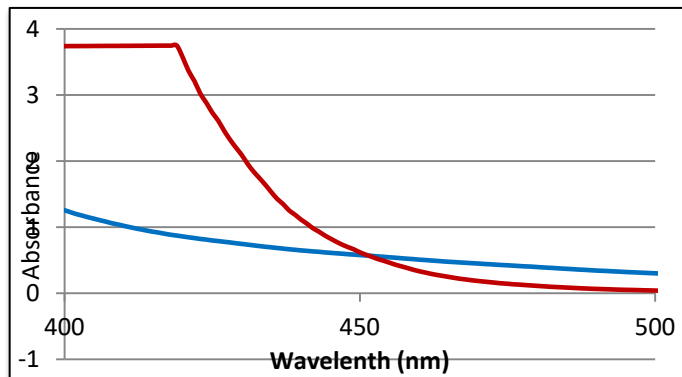
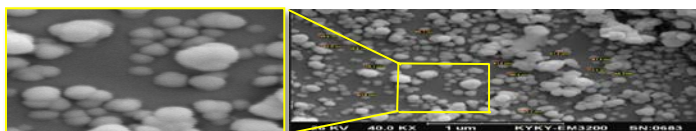


Fig 7. UV-VIS spectrum of peel Oak extract before (blue) and after (red) interacting with effluent fixer solution under optimized conditions

3.3.2. Particle size analysis

Fig 7 showed the Ag NPs were formed in mixtures. Therefore, for determination of particles size, dynamic light scattering is a powerful technique. It also able to assess dispersion size of particles. For this purpose, particle size analysis data was taken. (Fig 2a)

3.3.3. FT-IR studies

FT-IR analysis was used for the characterization of the extract and the synthesized nanoparticles (fig 8). The FT-IR spectra were recorded to identify the possible biomolecules responsible for the reduction of the Ag^+ ions and capping of the bio-reduced Ag-NPs recovered by the peel Oak extract. The FT-IR spectra of the peel Oak extract before (fig 8a) and after (fig 8b) interacting with effluent fixer solution have showed significant changes. In Fig 8b, broad peak in 3415 cm^{-1} can be allocated to the stretching vibration of the alcoholic hydroxyl group and Phenolic derivatives in the peel Oak extract. Other peaks may be attributed to the presence of stretching and bending vibrations of ethers, phenyl rings and C=C. Another peak at 1616 cm^{-1} is attributed to the stretching vibration of C=C functional groups and became shorter after synthesis of Ag-NPs. Therefore, the FT-IR results imply that the Ag-NPs successfully synthesized and capped with bio-compounds present in the peel Oak extract by using green method.

3.3.4. Morphology studies

In addition to particle size analyzer Scanning electron microscopy (SEM) also can be determined particle size. More than this SEM images are used for morphological characterization at the nanometer [51]. SEM analysis shows uniformly distributed silver nanoparticles that indicating the stabilization of nanoparticles by capping agents. (Fig 9)

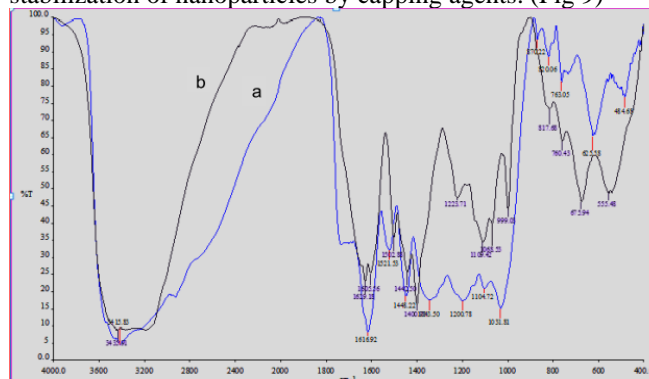


Fig 8. FT-IR spectrum of peel Oak extract before (a) and after (b) interacting with effluent fixer solution under optimized conditions

Fig 9. SEM image of Ag NPs obtained from interacted effluent fixer solution with peel Oak extract

SEM images of Ag NPs with an enlargement on its left side shows Ag NPs to be confined within a supporting matrix, likely comprised biomolecules such as phenolic compounds acting as a capping agent or stabilizer during synthesis, and thus controlling nanoparticle growth and clustering. Fig 9 were also shown the silver nanoparticles were spherical in shape and dispersity of particle size very close to results were acquired by other techniques in this research, previously and confirmed one.

3.3.5. XRD studies

X-ray diffractions used for the phase identification and characterization of the crystal structure of the silver nanoparticles. The crystalline nature of synthesized Ag NPs using peel Oak extract was confirmed from X-ray diffraction (XRD) patterns. (Fig 10)

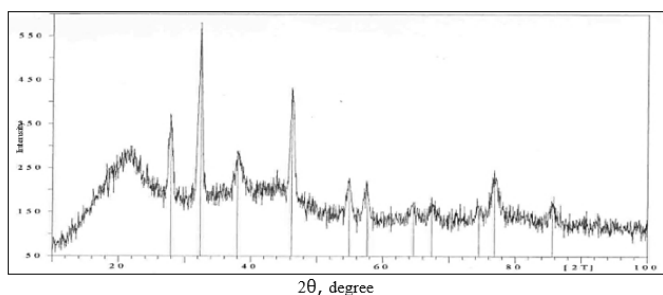


Fig 10. XRD pattern of Ag NPs synthesized by interaction of effluent fixer solution and peel Oak extract under optimized conditions

The XRD patterns of obtained nano particles of silver indicated that the structure of Ag NPs is face-centered cubic (fcc) [52]. Furthermore, the silver nanoparticles had a similar diffraction profile and the XRD peaks at 2θ of 38.12° , 44.34° , 64.23° , 77.21° and 81.47° can be indexed to the (1 1 1), (2 0 0), (2 2 0), (3 1 1) and (2 2 2) Bragg's reflections of the face-centered cubic (fcc) silver crystals, respectively [53]. Moreover, the XRD pattern shows the main crystalline phase is silver and other phases as impurities were not found in the XRD patterns (Ag XRD Ref. No. 01-087-0719). The particle size of the Ag-NPs formed were calculated using Debye - Scherrer equation that was almost 59.6 nm that were good in agreement with SEM and particle size analyzer results.

4. CONCLUSION

An interesting eco-friendly recovery of Silver nano-sized particles as a green recovery method has been developed. This method is a novel, environmental friendly and cost effective method that excludes the use of external stabilizing and capping agents and to the best, our knowledge this is the first report that applied effluent fixer solution as a precursor for producing of silver nano particles. The effects of various process variables like mixing ratio of effluent fixer solution to peel oak extract, pH, temperature, interaction time period and concentration were investigated. With mixing ratio of effluent to peel oak extract 30:1 in 70°C and pH 6 during 15 min of interaction time, nanoparticles below 62nm size with spherical shape and narrow dispersion was produced. The size of nano particles were determined with different techniques and the results of all of them in accordance with each other. FT-IR studies and SEM image showed the presence of bioorganic components which probable acted as a stabilizer for the synthesized silver nanoparticles.

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