APPLICATION OF DEBYE-SCHERRER FORMULA IN THE DETERMINATION OF SILVER NANO PARTICLES SHAPE

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ABSTRACT: Silver nano particles were synthesized by using green synthesis technique. Silver nano particles were characterized by u.v spectra, FTIR spectrum and XRD analysis. The shape of the silver nanoparticles was determined by using debye-scherrer formula. The particles shape was determined from the XRD analysis using the debye-scherrer formula.

Key words: green synthesis, silver nanoparticles, debye-scherrer formula, XRD, FTIR spectrum

I. INTRODUCTION

Paul Scherrer, proposed the Scherrer equation in 1981 [1]. This can be attributed to the fact that X-Ray diffraction is sensitive to the crystallite size inside the particles. From the well-known Scherrer formula the average crystallite size, L, is cos λK L (1)= θ β where λ is the X-ray wavelength in nanometer (nm), β is the peak width of the diffraction peak profile at half maximum height resulting from small crystallite size in radians and K is a constant related to crystallite shape, normally taken as 0.9. The value of β in 2θ axis of diffraction profile must be in radians. The θ can be in degrees or radians, since the cosθ corresponds to the same number.The Scherrer equation predicts crystallite thickness if crystals are smaller than 1000 Å or 100 nm.

Debye Scherrer equation for calculating the crystallite size is given by $\boldsymbol{D = K\lambda / \beta cos\theta}$ (1) where K is the Scherrer constant, λ is the wavelength of light used for the diffraction, β the "full width at half maximum" of the sharp peaks, and θ the angle measured. The Scherrer constant (K) in the above formula accounts for the shape of the particle and is generally taken to have the value 0.9. The results revealed that the crystallite size is less than 100 nm. , was developed in 1918, to calculate the nano crystallite size (L) by XRD radiationθ.cosβ λ =Scherrer Equation, L K of wavelength λ (nm) from measuring full width at half maximum of peaks (β) in radian located at any 2θ in the pattern. Shape factor of K can be 0.62 - 2.08 and is usually taken as about 0.89. But, if all of the peaks of a pattern are going to cosθ must be identical.βGive a similar value of L, then X-ray diffraction is a convenient method for determining the mean size of nano crystallites in nano crystalline materials.

The simplest way to obtain Scherrer equation is to take the derivation of Bragg's Law $\boldsymbol{2d\ sin\ \theta = n\lambda}$, Holding the wavelength λ constant and allowing the diffraction angle to broaden from a sharp diffraction peak from an infinite single crystal with perfect 3-dimeintinal order. For a single crystal, the diffraction from a set of planes with the distance d* occurs at a precisely θ*, so that λ = 2d* sinθ*. For many small nano crystals, diffraction from a lot of tiny crystals deviate ± Δθ from θ*. This means 2Δθ on the 2θ axis of diffraction pattern. The value of Δθ corresponds to FWHM or β, which is approximately half of 2Δθ. In other words since Δθ can be positive or negative, the absolute value must be taken and it reflects the half width of the shape line deviation in 2θ axis (full width at half maximum height, β). The derivation approach is taken by Alexander in Klug and Alexander "X-ray Diffraction" [2] to describe the Scherrer equation. It is also easily adoptable to describe the dependence of any two terms in the Bragg equation in terms of variability. Calculation of d-Spacing The value of d (the inter planar spacing between the atoms) is calculated using the Bragg's Law, $\boldsymbol{2d\ sin\ \theta = n\lambda}$ or $\boldsymbol{d = \lambda\ 2\ sin\theta(n=1)}$ $(\mathbf{2})$ Wavelength of X ray = 1.5 Å for CuKα.

The Bragg width contribution from crystallite size is inversely proportional to the crystallite size [3]. A portion of an unstrained grain appears in panel on the left, where the reflecting planes are equally spaced. If a uniform tensile strain is applied to a grain at right angles to the reflecting planes, their spacing becomes larger than d and the corresponding diffraction line shifts to lower angles but does not otherwise change. This line shift is the basis of the X-ray method for the measurement. The Bragg width is found to inversely proportional to the crystallite size [4]. When using the Debye-Scherrer equation for calculating particle size (D=Kλ/(β cos θ)Select a approiate peak that is not overlap with others, then be sure D and λ have the same unit (e.g. nm)FWHM is the full width at half maximum of the peak in rad,then you can calculate. You can calculate FWHM using origin software, and then debye-scherrer equation to calculate nanocrystallite size by using reference peaks.

II. EXPERIMENT

5 mL of aqueous extract of a green plant was taken in 100 mL volumetric flask separately and to this 95 mL of $AgNO_3$ solution was added. The same procedure was followed for all $AgNO_3$ concentrations. The flask was incubated at room temperatures. The colour change from pale yellow to dark brown was checked periodically. The reaction was followed by the study of formation of Ag-NP with the help of UV spectrum. The dark colour formation indicates that the silver nanoparticles .

The silver nanoparticle solution thus obtained was purified by repeated centrifugation at 5000 rpm for 20 min followed by redispersion of the pellet of silver nanoparticles into 10 ml of deionized water. After freeze drying of the purified silver particles, the structure and composition were analyzed by XRD. The crystallite domain size was calculated from the width of the XRD peaks, assuming that they are free from non-uniform strains, using the Scherr formula.

$D = 0.94 \lambda / \beta \cos \theta$ (1)

where D is the average crystallite domain size perpendicular to the reflecting planes, λ is the X-ray wavelength, β is the full width at half maximum (FWHM), and θ is the diffraction angle. To eliminate additional instrumental broadening the FWHM was corrected.This modified formula is valid only when the crystallite size is smaller than 100 nm.

III.DISCUSSION

The XRD analysis of synthesised AgNPs using stem of *Sterculia foetida* was recorded .The XRD patterns indicated that the structure of silver nanoparticles is face-centered cubic (fcc) [135]. In addition, the XRD peaks at 2θ of 38.17°, 44.31°, 64.44°, 77.34° (Fig-1) could be attributed to the 111, 200, 220, 311 data compared with JCPDS [146] crystallographic planes. The average nanocrystalline size has been estimated by using well known Debye–Scherrer formula, $D = k\lambda/\beta\cos\theta$, where D is particle diameter size, k is a constant equals 1, λ is wavelength of X-ray source (0.1541 nm), β is the full width at half maximum (FWHM) and θ is the diffraction angle corresponding to the lattice plane (1 1 1). The average crystallite size according to Debye–Scherrer equation calculated was found to be 5-11 nm. The colourless solution turned brown indicating the nanoparticle formation of silver. XRD analysis proves that silver nanoparticles were crystalline in nature. The characteristic brown color of silver provided a convenient spectroscopic signature to indicate nanoparticles formation. UV-spectra revealed maximum absorption peak at 440 nm and the intensity of absorption increased with time. The increase in intensity could be due to the increasing number of nanoparticles formed as a result of reduction of silver ions present in the aqueous solution with the help of phytochemicals present in the green plant.

Figure-1

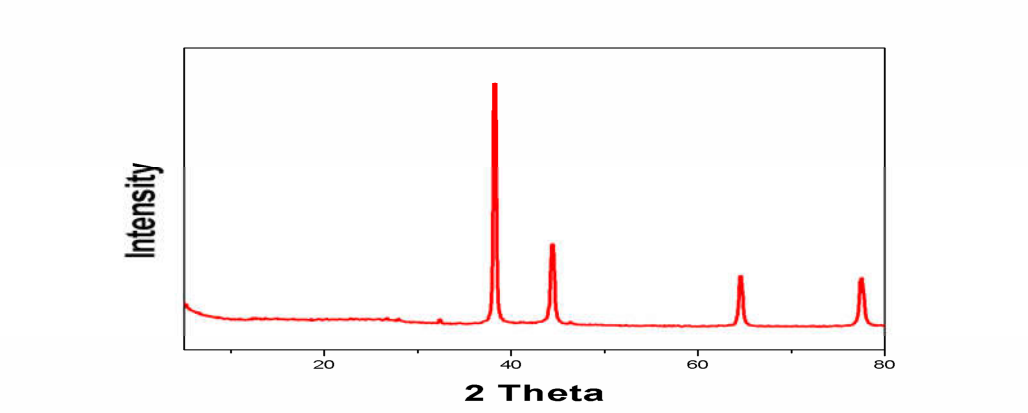

Table- 1

SAMPLE	FWHM	(110)PLANE ANGLE	(200)plane angle	(220)plane angle	(311)plane angle
SF-1	0.36144	38.4	44.5	64.7	77.6

IV. CONCLUSION

This study showed that the prepared silver nanoparticles were charecterised by XRD analysis .The shape of the silver nano particle was determined by debye-scherrer formula .The silver nanoparticles of plant sample SF-1 were in spherical in shape and the size of the silver nano particles were ranged from 5-11nm.

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