

# **PREPARATION AND CHARACTERIZATION OF POLYVINYL ALCOHOL-SILVER NANOPARTICLES COMPOSITE FILMS AND ITS ANTIMICROBIAL ACTIVITY**

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## **Abstract**

In this research work, silver nanoparticles were synthesized by using green synthesis. In green synthesis, neem leaf extract was used as reducing agent. Silver nanoparticles (SNP) were prepared by mixing neem leaf extract and 0.01 M AgNO<sub>3</sub> solution in the different ratios of 1:4, 2:4 and 3:4 v/v and the resulting silver nanoparticles were designated as SNP-NL1, SNP-NL2 and SNP-NL3 respectively. The existence of SNP in colloidal solutions was confirmed by Tyndall effect and UV-visible spectroscopy. The UV-visible spectrum revealed the formation of silver nanoparticles by exhibiting the typical surface plasmon absorption maxima at 415-420 nm. The silver nanoparticles after centrifugation of colloidal solution were characterized by modern techniques XRD, FT IR, SEM and EDXRF analyses. In XRD analysis, it was found that average crystallite size of SNP powders are in the range of 4.80 nm to 8.46 nm. From XRD analyses, all of the prepared SNP powders had the crystalline nature. The high intensity peaks of the prepared samples confirmed the diffraction faces of silver. According to the XRD spectra of all of the prepared SNP-NL, there was impurity peaks in the SNP-NL1 and SNP-NL3 but no impurity peaks found in the SNP-NL2. The crystallite size of SNP-NL2 was 6.93 nm. From the FT IR spectra of all of the prepared SNP-NL, it was observed that the stretching and bending vibration of residual organic functional groups from the leaf extract are present. SEM micrographs of all of the prepared SNP-NL showed agglomeration and larger particle size distribution. From EDXRF analyses, the main constituent element in the SNP-NL2 is Ag (92.384 %). The different types of polyvinyl alcohol (PVA) film were prepared by using different concentrations (1 - 5 % w/v) of PVA in distilled water. The obtained PVA films were designated as PVA-1, PVA-2, PVA-3, PVA-4 and PVA-5. According to the physicochemical properties, the optimum conditions of PVA-3 film has tensile strength (31.7 MPa), elongation at break (241 %) and tear strength (155.8 kNm<sup>-1</sup>). The selected PVA-3 film was characterized by XRD, SEM, FT IR and TG-DTA analyses. The PVA-

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SNP composite films were prepared by varying the volume ratios of 3 % w/v PVA solution and colloidal SNP-NL2 solution. The antimicrobial activity of the prepared PVA-SNP composite films was investigated by using agar well diffusion method.

**Keywords:** Neem leaf extract, silver nanoparticles, green synthesis, PVA-SNP composite films, antimicrobial activity

## Introduction

Nanoparticles are fundamental building blocks of nanotechnology. The most important and distinct property of nanoparticles is their larger surface area to volume ratio. Nanoparticles (NPs) are usually clusters of atoms in the size range of 1-100 nm (Lalitha, 2013). The properties of a metal NP are determined by its size, shape, composition, crystallinity, and structure. Silver nanoparticles (SNPs) have a number of application from electronics and catalysis to infection prevention and medical diagnosis. SNPs has been known as excellent antimicrobial and anti-inflammatory agents and were used to improve wound healing. A number of physical and chemical strategies were employed for the synthesis of SNPs (Sivakumar, 2012).

An eco-friendly green mediated synthesis of inorganic nanoparticle is a fast growing research in the limb of nanotechnology. The biosynthesis method employing plant extract have drawn attention as a simple and viable alternative to chemical procedures an physical methods. Bioreduction of silver ions to yield metal nanoparticles using living plants, geranium leaf, neem leaf have been studied. *Azadirachta indica* is one of the most versatile medicinal plant having a wide spectrum of biological activity. Every part of the tree has been used as a traditional medicine for household remedy againt various human ailment, from antiquity.

*Azadirachta indica* leaf extract has been utilized for the synthesis of silver nanoparticles. The major advantage of using the neem leaves is that it is a commonly available medicinal plant and the antibacterial activity of the biosynthesized silver nanoparticle might have been enhanced as it was capped with the neem leaf extract (Lalitha, 2013). *Azadirachta indica* leaf extract is used in the synthesis of various particles like gold, zinc oxide and silver etc. The phytochemicals present in neemleaf are namely terpenoids and flavonoids, which act as reducing agent as well as capping agent and helping

the stabilizing the nanoparticles. When silver salt is treated with neem leaf extract, the silver salt is reduced to SNPs (Verma, 2016).

Silver nanoparticles exhibit distinct optical activities that have found wide use in electronics, catalysis and in sensing based applications. Moreover, it displays antimicrobial activity against a broad spectrum of bacteria and fungi and thus finds use as a biocide and also in the preparation of bactericidal nanomaterials for wound dressings and surgical purposes. Silver nanoparticles are non-toxic to humans in low concentrations. The silver-nanoparticles can inactivate proteins, blocking respiration and electron transfer (Albrecht *et al*, 2006).

Polyvinyl alcohol (PVA) is a bio- friendly polymer as it is water soluble and has extremely low cytotoxicity. PVA belongs to the group of polymers which can be used in combination with silver nitrate. PVA is one of the synthetic, biodegradable, biocompatible polymer utilized in medical applications such as wound dressing, artificial skin, coatings, transdermal patches, cardiovascular devices and drug delivery systems (Sayed, 2014). In this research work, PVA-SNP composite films are investigated against strains of different bacteria.

## **Materials and Methods**

### **Sample Collection**

Neem leaves were collected from Pyay Township, Bago Region. The collected leaves were rinsed several times with running tap water and after that with distilled water. Then it was air- dried at room temperature.

### **Preparation of *Azadirachtaindica* A. Juss (Neem Leaf) Extract**

Crudeneem leaf extract was prepared by taking 25 g of *Azadirachtaindica* leaves in a 500 mL Erlenmeyer flask with 200 mL of deionized water and then boiled the mixture for one hour on water bath. The sample solution was filtered and cooled at room temperature.

### **Green Synthesis of Silver Nanoparticles (SNPs)**

Silver nanoparticles were prepared by mixing neem leaf extract and 0.01 M AgNO<sub>3</sub> solution in the ratios of 1:4, 2:4, 3:4 v/v in a 250 mL

Erlenmeyer flask. Dilute ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) solution was used to maintain the pH of the reaction mixture in the range of 8-9. The flask was kept for 2 h in magnetic stirrer at 80 rpm to achieve homogeneous reaction. Sonification was carried out to reduce size and purify the silver nanoparticles in the colloidal solution. The solution containing silver nanoparticles were centrifuged at 7000 rpm for 20 min. The purified particles were dried by using a hot air oven up to  $70^\circ\text{C}$  for one and half hours. And then solid silver nanoparticles were obtained.

### **Confirmation for the Existence of Silver Nanoparticles in Solution by Tyndall Effect**

The laser pointer was placed to the edge of the bottle containing SNP colloidal solution and the light was passed through the solution. The photograph of observation about the existence of silver nanoparticles in solution was presented in Figure 1.

### **Characterization of Silver Nanoparticles**

UV-visible spectrophotometer model is SHIMADZU (UVmini-1240, JAPAN). UV spectra of the silver colloid in the range 330 nm - 460 nm were measured. UV absorption spectra have proved to be quite sensitive to the formation of silver colloids because silver nanoparticles exhibit an intense absorption peak due to the surface plasmon excitation. The absorption band in visible light region (350 nm- 550 nm) is typical for silver nanoparticles. The plasmon peak and the full-width at half-maximum (FWHM) depend on the extent of colloid aggregation.

The phase identification of the silver nanoparticles was carried out by X-ray diffraction method. The solid sample was grounded using a mortar and pestle into powder. X-ray powder diffraction measurement was carried out by using (Rigaku, Miniflex-600) powder diffractometer with long fine focus Cu anode.

FT IR measurements were carried out to identify the biomolecules for capping and efficient stabilization of the metal nanoparticles synthesized. The samples were measured by using Perkin Elmer GX System, FT IR spectrophotometer.

Morphology of the silver nanoparticles were observed on JSM 5610 LV Scanning Electron Microscopy, JEOL-Ltd., Japan.

Elemental compositions in the prepared silver nanoparticles were determined by EDXRF using EDX-8000 spectrometer (Shimadzu Co.Ltd., Japan).

### **Preparation of Pure Polyvinyl Alcohol Films (PVA)**

Polyvinyl alcohol (PVA) films were prepared by solution casting method. Different concentrations of PVA (molecular weight 14,000, degree of hydrolysis 98 %) (1, 2, 3, 4 and 5 % w/v) solution were prepared with distilled water by stirring and heating at 50 °C. The PVA solutions were placed in an autoclave at 0.1 MPa and 121°C for 20 min. Each polymer solution was casted on melamine plate and dried in air. The series of PVA films were obtained.

### **Preparation of Polyvinyl Alcohol- Silver Nanoparticles Composite Films (PVA-SNP)**

Polyvinyl alcohol- silver nanoparticles composite films were prepared by mixing different volume ratios of 3 % (w/v) of PVA solution and the prepared SNP-NL2 solution (95:5, 90:10, 85:15, 80:20, 75:25, 70:30 v/v) to make up 100 mL. The mixed solutions were stirred by using a magnetic stirrer at 80 rpm for 20 min. Then polymer solutions were kept for sufficient time to remove any bubble formation. Each polymer solution was placed on melamine plate and dried in air. The melamine plates containing the composite solutions were left about 7 days to obtain PVA-SNP composite films. The composite films after drying were removed easily from the melamine plates.

### **Determination of the Antimicrobial Activity by Agar Well Diffusion Method**

The PVA-SNP composite films were tested with (a) *Bacillus subtilis* (b) *Staphylococcus aureus* (c) *Pseudomonas aeruginosa* (d) *Bacillus pumilus* (e) *Candida albicans* (f) *Escherichia coli* species to investigate the nature of antimicrobial activity.

## Results and Discussion

### Biosynthesis of Silver Nanoparticles by Using Neem Leaf Extract as Reducing agent

Different volumes of neem leaf extract were mixed with 0.01 M  $\text{AgNO}_3$  solution in three different ratios of 1:4, 2:4 and 3:4 v/v without varying the other conditions. Reduction of the silver ions to silver nanoparticles during exposure to the plant leaf extract was followed by colour change from pale yellow to reddish brown colour. This is due to the excitation of surface plasmon vibration in silver nanoparticles.

### Tyndall Effect

Tyndall effect on silver nanoparticles (SNPs) is shown in Figure 1. It was found that the laser light passes through the solution due to the presence of nanoparticles.

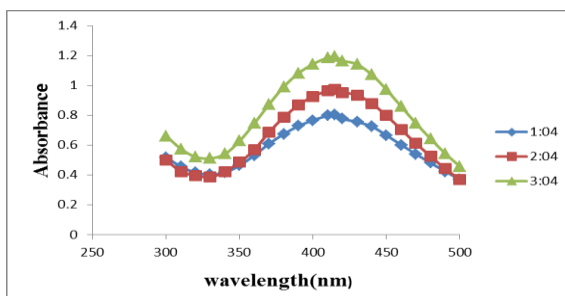


**Figure 1:** Tyndall effect on the prepared SNPs by green synthesis

### Characterization of Silver Nanoparticles

#### UV-visible Studies

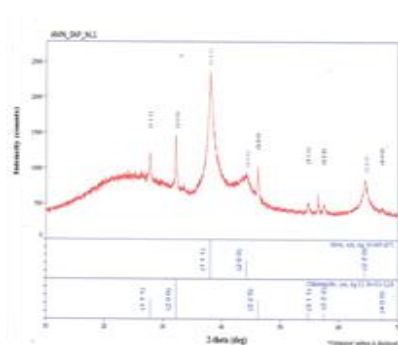
The sample when treated with complete reaction conditions, change in colour of extracts suspension from pale yellow to brownish red was observed. This colour change preliminary showed the presence of silver nanoparticles or reduction of  $\text{Ag}^+$  of  $\text{AgNO}_3$  to  $\text{Ag}^0$ . After observing changes in colour of the extracts, the maximum absorbance was observed at 415 nm due to surface resonance of silver nanoparticles shown in the Figure 2.



**Figure 2:**UV-visible spectra of prepared silver nanoparticles (SNP-NL)by green synthesis

**X Ray Diffraction Studies**

The XRD data were obtained in the  $2\theta$  range from  $10^\circ$  to  $70^\circ$  in step scan mode with  $2\theta$  step of  $0.02^\circ$ . The diffraction pattern indicated that the sample is the silver nanoparticles. The conversion of silver nitrate to silver nanoparticle was greater than ninety percent and smaller peaks contributed to neem extract impurity. The XRD pattern of SNPs is shown in the Figure 3 (a, b, c). From XRD analysis, all prepared silver nanoparticles give (111), (200) and (220) reflection planes between  $2\theta$  values  $30^\circ$ - $70^\circ$  and cubic crystal structure. However, SNP-NL2 samples show only single phase of Ag and no impurity peaks. Therefore, SNP-NL2 was chosen for the optimum sample. The crystallite sizes of all of the prepared SNP- powders were calculated by Debye-Scherrer equation in Table 1 (a, b, c). According to Table 2, the average crystallite size of the prepared SNP powders are SNP-NL1 (8.46 nm), SNP-NL2 (6.93 nm) and SNP-NL3 (4.8 nm).

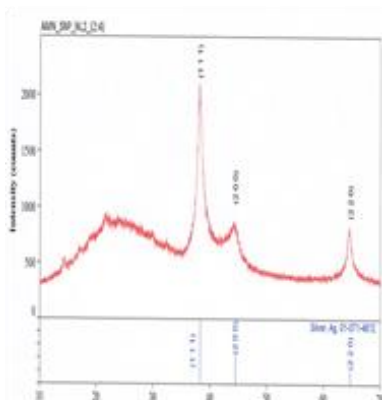


**Figure 3:(a)** XRD diffractogram of silver nanoparticles by green synthesis SNP-NL1

**Table1(a)Crystallite Size of Silver Nanoparticles by XRD**

$2\theta$	FWHM	(hkl)	$\square$ ( $\text{\AA}$ )	Crystalline size (nm)
37.919	1.035	111	1.5406	8.48
44.050	1.910	200	1.5406	4.70
64.420	0.810	220	1.5406	12.20
<b>Average</b>				<b>8.46</b>

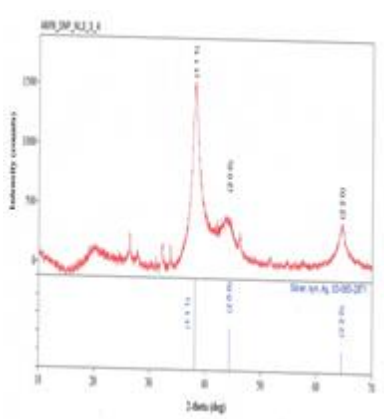
(SNP-NL1)



**Figure3:(b)** XRD diffractogram of silver nanoparticles by green synthesis SNP-NL 2 (SNP-NL2)

**Table 1: (b) Crystallite Size of Silver Nanoparticles by XRD**

$2\theta$	FWHM (hkl)	$\square$ ( $\text{\AA}$ )	Crystalline size (nm)
38.259	1.060	111	8.28
44.210	2.070	200	4.32
64.540	1.190	220	8.20
Average			6.93



**Figure3:(c)** XRD diffractogram of silvernanoparticles by green synthesis SNP-NL 3

**Table 1: (c) Crystallite Size of Silver Nanoparticles by XRD**

$2\theta$	FWHM (hkl)	$\square$ ( $\text{\AA}$ )	Crystalline size (nm)
38.170	1.70	111	5.15
44.080	3.13	200	2.86
64.510	1.54	220	6.39
Average			4.80

(SNP-NL3)

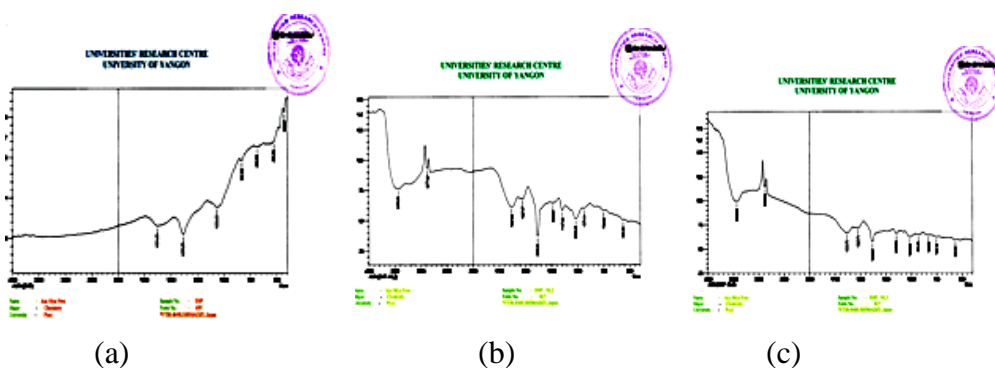
**Table2: Average Crystallite Size of the Prepared SNP Powder Using Green Synthesis by XRD Analysis (0.01 M  $\text{AgNO}_3$ )**

Samples	NL : $\text{AgNO}_3$ (v/v)	Crystallite size (nm)	Crystal structure
SNP-NL1	1 : 4	8.46	Cubic
SNP-NL2	2 : 4	6.93	Cubic
SNP-NL3	3 : 4	4.80	Cubic



## FT IR Analysis

Figure 4 shows the FT IR spectra of all of the prepared SNP-NL, the characteristic absorption bands at 3435, 2877, 1631, 1018  $\text{cm}^{-1}$  were observed. These peaks correspond to groups present in the sample and are indicated to O-H stretching, C-H stretching, C=C stretching and C-O-C stretching which is the good correlation with that of literature. These bands were confirmed the presence of terpenoids and flavonoids in neem leaf. It was inferred that terpenoids present in neem leaf extract acts as stabilizing as well as capping agents. From FT IR spectrum of SNP-NL, it was observed that the carbonyl group from amino acid residues and proteins could possibly for the metal nanoparticles to prevent agglomeration and stabilize the medium. The biological molecules were performed dual functions of formation and stabilization of silver nanoparticles in the aqueous medium. Table 3 shows FT IR spectral peaks of SNP-NL1, SNP-NL2 and SNP-NL3.



**Figure 4:** FT IR spectra of the prepared SNP-NL (a) SNP-NL1, (b) SNP-NL2 and (c) SNP-NL3

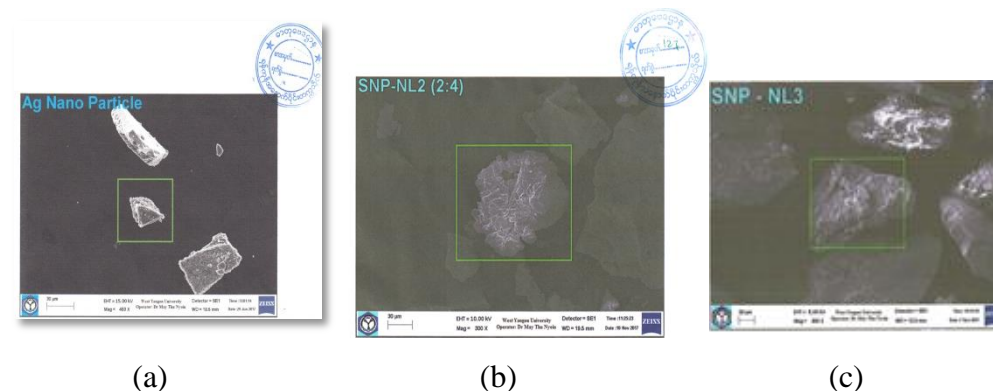
**Table 3: FT IR Spectral Assignments of the Prepared Silver Nanoparticles**

Experimental Frequency (cm <sup>-1</sup> )			* Literature Frequency (cm <sup>-1</sup> )	Band Assignments
SNP-NL1	SNP-NL2	SNP-NL3		
-	3435	3435	3600-3000	-OH stretching vibration
-	2877	2875	2980-2800	C-H stretching vibration of sp <sup>3</sup> hydrocarbons
1631	1631	1633.7	1620-1580	C=C ring skeletal stretching vibration
1386	1383	1383.1	1380-1300	-OH bending vibration
1068	1018	1016.5	1100-1025	C-O-C stretching vibration
831,526,428	565	833,567	830-500	C-H out of plane bending vibration

\*Silverstein, (1998)

### SEM Analysis

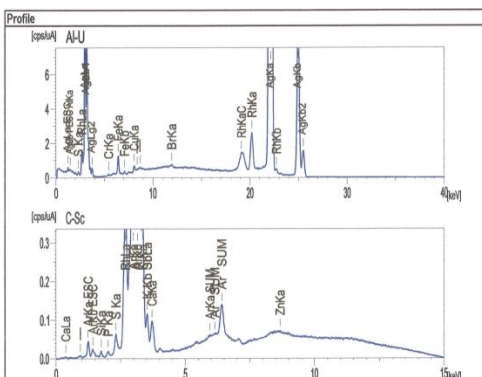
Further characterization of silver nanoparticles was done by using scanning electron microscope (SEM). The scanning electron micrographs of the prepared silver nanoparticles are shown in Figure 5. It can be concluded that SNPs are initially monodispersed but drying process lead to agglomeration of many particles resulted into larger size particles.



**Figure 5:** Scanning electron micrograph of silver nanoparticles by green synthesis (a) SNP-NL1, (b) SNP-NL2 and (c) SNP-NL3 a = 403 X magnification, b = 300 X magnification, c = 300 X magnification

**EDXRF analysis**

Figure 6 shows EDXRF spectra of SNP-NL2. According to the EDXRF spectra of the prepared SNP-NL2, silver were major constituent (92.384 %) and other were trace constituents. Table 4 shows the relative abundance of elements in the prepared SNP-NL2 by EDXRF.



**Figure 6 :**EDXRF spectra of SNP-NL2

**Table 4:Relative Abundance of Elements in Prepared SNP-NL2 by EDXRF**

No.	Elements	Relative Abundance (%)
1	Silver	92.384
2	Aluminum	2.791
3	Potassium	1.503
4	Silicon	0.911
5	Calcium	0.808
6	Sulphur	0.532
7	Iron	0.532
8	Phosphorus	0.325
9	Copper	0.108
10	Chromium	0.075
11	Bromine	0.031

**Table 5:Yield Percentage of Silver Nanoparticles**

No	Sample	Weight of Silver in Silver nitrate (g)	Weight of Silver Particles (g)	Yield (%)
1	SNP-N 1	0.17	0.050	46.30
2	SNP-N 2	0.17	0.062	57.41
3	SNP-N 3	0.17	0.052	48.15

According to the Table 5, the yield percentage of silver nanoparticles was found to be SNP-NL1 (46.30 %), SNP-NL2 (57.41 %) and SNP-NL3 (48.15 %). Among them, SNP-NL2 gave more silver nanoparticles.

### Aspect of the Preparation of Pure PVA Film

Pure PVA films were prepared using various percents of PVA (1 % to 5 % w/v) in distilled water by solution casting method. The prepared PVA films were designated as PVA-1, PVA-2, PVA-3, PVA-4 and PVA-5 according to the percent of PVA. The prepared pure PVA films appeared to be homogeneous, transparent and colourless. According to the physicomechanical properties (tensile strength, elongation at break and tear strength) of prepared PVA films, PVA-3 was chosen for the optimum films according to tensile strength, elongation at break and tear strength as shown in Figure 7 and Table 6.



**Figure 7:** The photographs of (a) PVA-1 (b) PVA-2 (c) PVA-3 (d) PVA-4 (e) PVA-5 films

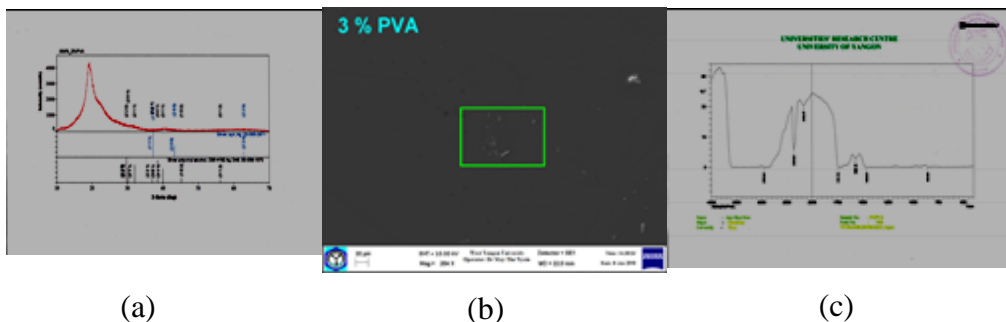
**Table 6: Physicomechanical Properties of the Prepared Polyvinyl Alcohol Films**

Prepared Films	PVA(%) w/v	Tensile Strength (MPa)	Elongation at Break(%)	Tear Strength ( $\text{kNm}^{-1}$ )
PVA-1	1	26.0	128	96.3
PVA-2	2	29.7	202	114.0
PVA-3	3	31.7	241	155.8
PVA-4	4	27.1	216	87.9
PVA-5	5	33.0	282	101.0

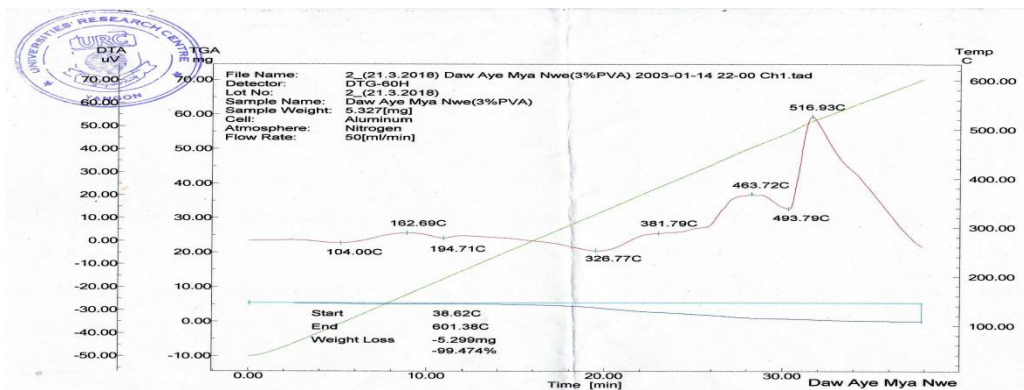
Thickness = ~ 0.57 mm

### Characterization of the Prepared PVA Film

The selected prepared PVA-3 film was characterized by modern techniques such as XRD, SEM, FT IR were shown in Figure 8. The XRD pattern of PVA film exhibits a broad diffraction peak due to the amorphous nature of the polymer. The SEM micrograph of the prepared PVA-3 membrane has smooth surface and homogeneous film. The FT IR spectrum of pure PVA film exhibits a major peaks associated with PVA. The major peaks were observed in the  $3600\text{ cm}^{-1}$ ,  $2955\text{ cm}^{-1}$ ,  $1568\text{ cm}^{-1}$  and  $1458\text{ cm}^{-1}$ . These major peaks showed the O-H stretching, C-H stretching, C=C stretching and O-H bending. As seen in Figure 9, the thermogram of PVA-3 film possesses three stages of distinct weight loss between  $38\text{ }^{\circ}\text{C}$  to  $600\text{ }^{\circ}\text{C}$ . The first stage ranged between  $38\text{ }^{\circ}\text{C}$  and  $120\text{ }^{\circ}\text{C}$  with  $11.04\%$  of weight loss and this was due to the evaporation of loosely bound water. The second stage ranged between  $120\text{ }^{\circ}\text{C}$  and  $350\text{ }^{\circ}\text{C}$  was due to the scission of functional group of polymer chain. The third stage of weight loss indicated the degradation of polymer backbone and progressive rupture of chain, combustion and formation of residue.



**Figure 8:** (a) XRD diffractogram, (b) SEMmicrograph and (c) FT IR spectrum of the prepared polyvinyl alcohol PVA-3 film



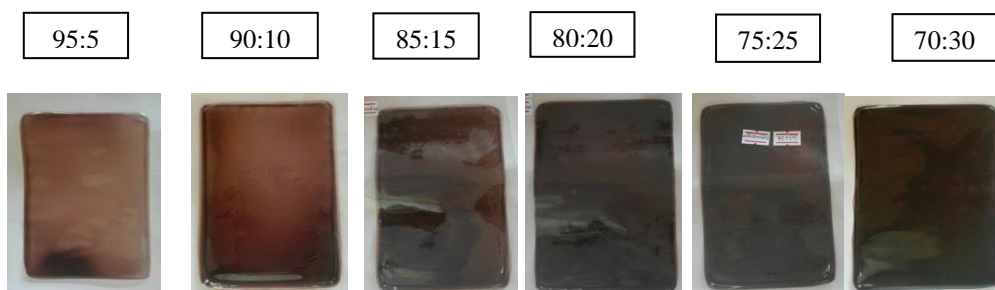
**Figure 9:** TG-DTA thermogram of the prepared polyvinyl alcohol PVA-3 film

**Table 7: Thermal Analysis Data of the Prepared Polyvinyl Alcohol PVA-3 Film**

TG Thermogram			Nature of Peak DTA	Remark
Temperature Range ( $^{\circ}$ C)	Total Weight Loss (%)	Break in Temperature ( $^{\circ}$ C)		
38-120	11.04	104	endothermic	due to the evaporation of loosely bound water
120-350	33.13	326	endothermic	due to the scission of functional group of polymer chain
350-600	55.30	463 516	Exothermic	Due to the degradation of polymer backbone and progressive rupture of the chain, combustion and formation of residue

### Aspect of Preparation of PVA-SNP Films

The PVA-SNP composite films were prepared by solution casting method from solutions of PVA-3 and SNP-NL2 in deionized water at various compositional ratios. The basic method for the synthesis of NPs in PVA is to disperse metal ion solution in the polymer and reduce to zero valent states. The PVA-SNP composite films were prepared by using different volume ratios of PVA-3 solution and SNP-NL2 colloidal solutions (95:5, 90:10, 85:15, 80:20, 75:25 and 70:30). The obtained composite films were designated as PVA-SNP-1, PVA-SNP-2, PVA-SNP-3, PVA-SNP-4, PVA-SNP-5 and PVA-SNP-6 respectively. The effect of composite films composition on mechanical properties and antimicrobial activity were studied. Solutions of PVA-SNP appeared to be homogeneous and transparent. The colour of the solution varied from colourless of pure PVA solution to dark brown colour with increasing SNP content. The distinctive colours of nanosilver are due to the phenomenon known as plasmon absorbance. Incident light creates oscillations in conduction electrons on the surface of the NPs and electromagnetic radiation is absorbed. This indicates the formation of AgNPs. With an increase in reaction time, particle size and aggregation of silver nanocrystal gradually increased together. After evaporation of the solvent, the prepared films of PVA-SNP composite films were found to be transparent. Figure 10.



(a) PVA-SNP-1 (b)PVA-SNP-2 (c)PVA-SNP-3 (d) PVA-SNP-4 (e) PVA-SNP-5 (f) PVA-SNP-6

**Figure 10:** The photographs of PVA-SNP composite films with various volume ratios of PVA-3 solution and colloidal SNP solution

### Aspect of Physicomechanical Properties of Polyvinyl Alcohol- Silver Nanoparticles Composite Films

The physicomechanical properties of polyvinyl alcohol-silver nanoparticles composite films were shown in Table 8. From the resulting data, PVA-SNP- 3 composite film was found that tensile strength of 30.8 MPa, elongation at break of 231 % and tear strength of 117 kNm<sup>-1</sup>. Therefore, PVA-SNP-3 was chosen for optimum film due to its highest elongation at break.

**Table 8: Physicomechanical Properties of Polyvinyl Alcohol- Silver Nanoparticles composite Films**

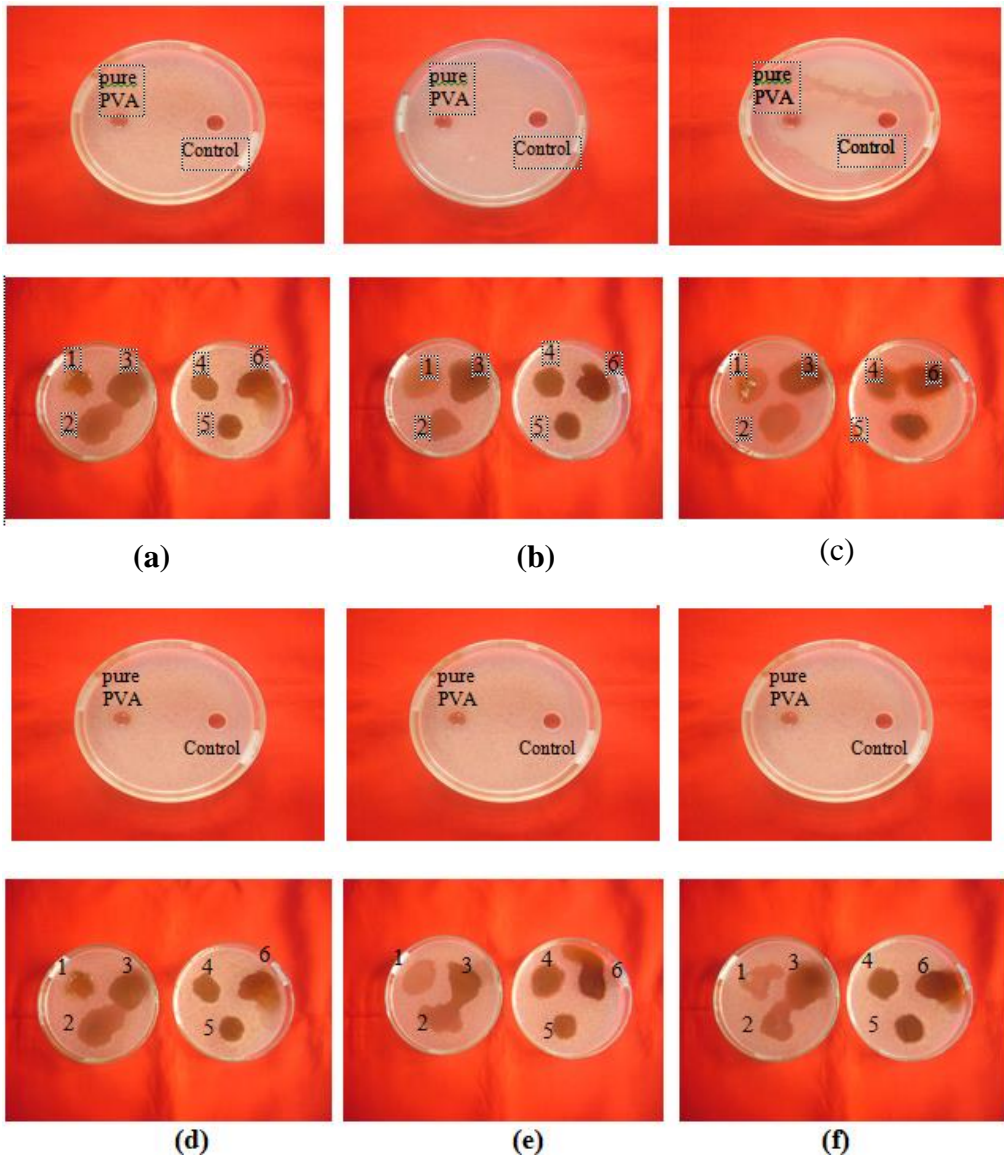
No.	Parameters	PVA-SNP Composite Films					
		PVA-SNP-1	PVA-SNP-2	PVA-SNP-3	PVA-SNP-4	PVA-SNP-5	PVA-SNP-6
1	Tensile strength (MPa)	3.0	26.1	30.8	29.2	29.7	33.3
2	Elongation at Break (%)	133	147	231	168	89	221
3	Tear Strength(kNm <sup>-1</sup> )	16.7	152.7	117.0	128.7	56.0	123.0

Thickness = ~ 0.43 mm

### Antimicrobial Activity of PVA-SNP Composite Films

Silver is known for its antimicrobial properties and has been used for many years in the medical field for antimicrobial applications. Additionally, silver has been used in water and air filtration to eliminate microorganisms. Inhibition zone values were obtained from the synthesized PVA, PVA-SNP composites tested against six microorganisms: (a) *Bacillus subtilis* (b) *Staphylococcus aureus* (c) *Pseudomonas aeruginosa* (d) *Bacillus pumilus* (e) *Candida albicans* (f) *Escherichia coli*. Antimicrobial activity of PVA-SNP composite films has been investigated by agar well diffusion method as shown in Figure 11 and Table 9. It was observed that the prepared PVA-3 film did not show antimicrobial activity, however PVA-SNP composite films showed the antimicrobial activity.





**Figure 11:** Antimicrobial activity of the prepared (1) PVA-SNP-1, (2) PVA-SNP-2, (3) PVA-SNP-3, (4) PVA-SNP-4, (5) PVA-SNP-5 and (6) PVA-SNP-6 composite films (a) *Bacillus subtilis* (b) *Staphylococcus aureus* (c) *Pseudomonas aeruginosa* (d) *Bacillus pumilus* (e) *Candida albicans* (f) *Escherichia coli*

**Table 9: Antimicrobial Activity of the Prepared Polyvinyl Alcohol-Silver Nanoparticles Composite Films by Agar Well Diffusion Method**

Sample Films	Inhibition zone diameters of the samples against six microorganisms (mm)					
	(a) <i>Bacillus subtilis</i>	(b) <i>Staphylococcus aureus</i>	(c) <i>Pseudomonasaeruginosa</i>	(d) <i>Bacillus pumilus</i>	(e) <i>Candida albicans</i>	(f) <i>Escherichia Coli</i>
Pure PVA	-	-	-	-	-	-
PVA-SNP-1	15 mm	19 mm	17 mm	15 mm	16 mm	16 mm
PVA-SNP-2	19 mm	18 mm	18 mm	18 mm	17 mm	17 mm
PVA-SNP-3	18 mm	19 mm	19 mm	19 mm	17 mm	19 mm
PVA-SNP-4	17 mm	17 mm	16 mm	16 mm	17 mm	15 mm
PVA-SNP-5	15 mm	15 mm	15 mm	15 mm	15 mm	15 mm
PVA-SNP-6	17 mm	17 mm	16 mm	16 mm	16 mm	16 mm

Agar Well 10 mm (-), 10 mm ~14 mm (+), 15 mm ~19 mm (++), 20 mm ~above (+++)

## Conclusion

In this research work, the silver nanoparticles were synthesized from neem leaf extract by green synthesis. The synthesized silver nanoparticles were characterized by UV-visible spectroscopy, FT IR, SEM, EDXRF and XRD analysis. By the determination of UV-visible spectra, the maximum absorption peak of colloidal silver nanoparticles were appeared at 415 nm. FT IR spectrum of SNP-NL indicated the absorption bands at 3435, 2877, 1631 and 1018  $\text{cm}^{-1}$ . The absorption band at 3435  $\text{cm}^{-1}$  is corresponding to O-H stretching, 2877  $\text{cm}^{-1}$  is due to C-H stretching, 1631  $\text{cm}^{-1}$  is due to C=C stretching and 1018  $\text{cm}^{-1}$  is due to C-O-C stretching. The band at 565  $\text{cm}^{-1}$  is corresponding to C-H out of plane bending that is responsible for reducing the  $\text{Ag}^+$  to  $\text{Ag}^0$ . From XRD analysis, the average crystallite sizes of all of the prepared SNP-NL were 8.46 nm (SNP-NL1), 6.93 nm (SNP-NL2) and 4.80 nm (SNP-NL3). According to XRD spectra of all of the prepared SNP-NL, there was no impurity peaks in the SNP-NL2. From SEM analysis, all of the prepared SNP-NL were initially monodispersed but drying process caused agglomeration of many particles resulted into larger size particles. According to EDXRF spectra of the prepared SNP-NL2, silver were major constituent (92.384 %) and other were trace constituents. The yield percentage of all of the prepared SNP powders were 46.30 % (SNP-NL1), 57.41 % (SNP-NL2)

and 48.15 % (SNP-NL3). Among them, SNP-NL2 gave more silver nanoparticles. The pure PVA films were prepared by varying different weight percents of 1-5 % w/v PVA solution by using solvent evaporating method. According to the mechanical properties of PVA films, PVA-3 film was chosen for the preparation of PVA-SNP composite film. The characterization by modern techniques such as XRD, SEM, FT IR and TG-DTA were able to reveal the surface morphological texture, pronounced functional groups as well as thermal stabilities of the prepared films. According to the TG-DTA thermogram of the prepared PVA-3 film, three stages of weight loss were observed. These weight loss were due to the evaporation of loosely bound water, the scission of functional group of polymer chain and the degradation of polymer backbone and progressive rupture of the chain, combustion and formation of residue. According to the physicomechanical properties of the prepared PVA-SNP composite film, PVA-SNP-3 has optimum tensile strength (30.8 MPa), elongation at break (231 %) and tear strength ( $117 \text{ kNm}^{-1}$ ). Although the prepared PVA-3 film did not show the antimicrobial activity, PVA-SNP composite films were observed to exhibit the antimicrobial activity against all of the tested microorganisms.

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