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Characterisation of Xanthan Gum Silver-Mediated Nanoparticles Synthesized from Xanthan Gum Produced by Xanthomonas SP Strain Sfl2 Using Sweet Potato Peel

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Abstract: Xanthan gum [$(C_{35}H_{49}O_{29})n$] is a microbial exopolysaccharide which consists of pentasaccharide repeat units comprising of glucose, mannose and glucuronic acid. Xanthan gum is a biopolymer and as such it can act as both reducing and capping agents in metal nanoparticle synthesis.Nanoparticles are small particles that range between 1 to 100 nanometres in size; they have distinctive properties such as high surface area to volume ratio with myriad activities applicable in a number of applied sectorshowever chemical synthesis of these nanoparticles involves the use of hazardous substances which are toxic to humans and the environment. Hence, the need to synthesis nanoparticles using eco-friendly, cost effective and biocompatiblemethod.Silvernanoparticle was synthesised from xanthan gumproduced by Xanthomonas sp strain sfl2 using sweet potato peel with 9Mm of sliver nitrate ($AgNO_3$). The xanthan gum silver-mediated nanoparticles (XGNSp) synthesised was characterised using UV-Vis spectrophotometry (UV-VIS), Scanning Electron Microscopy Energy Dispersion (SEM-EDX), Fourier Transform Infrared spectroscopy (FTIR),Transmission Electron Microscopy (TEM) and X-ray Diffraction (XRD).The UV-Vis indicated XGSNp synthesis at 418.724 nm. FTIR showed peaks between 678.4 and 3280.1 cm^{-1} indicating aldehydes, hydroxyl and carbonyl groups. SEM revealed XGSNp to be fibrous homogenous structures, EDX revealed it contained silver (Ag), carbon (C), oxygen (O),TEM showed XGSNp as spherical shape with size range from 10.40 to 15.89 nm and X-ray diffraction presented XGSNp to be crystalline in nature.The study concluded that xanthan gum silver-mediated nanoparticles can be synthesised using green synthesis and these synthesised nanoparticles have characteristics significant for various food and industrial applications.

Keywords: Exopolysaccharides, nanoparticles, silver nanoparticle, xanthan gum, xanthan gum silver-mediated nanoparticle,UV-Vis spectrophotometry (UV-VIS), Scanning Electron Microscopy Energy Dispersion (SEM-EDX), Fourier Transform Infrared spectroscopy (FTIR), Transmission Electron Microscopy (TEM) and X-ray Diffraction (XRD).

Introduction

Nanoparticles (NPs) are small particles that range between 1 to 100 nanometres in size;they have distinctive properties which can be rightfully manipulated for several preferred applications due to their high surface area to volume ratio and vast electronic, optical, magnetic, chemical, and physical properties (Khan et al., 2017). They are considered most important due to their unique properties and their ability to form diverse nanostructures. Nanoparticles can be synthesized usingBottom-up approach and Top-down approach. In the bottom up approach, NPs are formed fromrelatively simpler substances. Examples of this case are sedimentationand reduction techniques while the Top-down approachstartsfrom larger molecule, which decomposed into smaller units and then these units, are converted into suitable NPs (Baig et al., 2021).NPs are not simple molecules itself and therefore composed of three layers i.e. the surface layer (which may be functionalized with a variety of small molecules, metal ions, surfactants and polymers), the shell layer (which is chemically different material from the core in all aspects), and the core (which is essentially the central portion of the NPs and usually refers the NP itself) (Shin et al., 2016). Nanoparticles can be classified into carbon based, ceramic, lipid based, semi-conductor, polymeric and metal nanoparticles. Metal NPs are purely made of the metals precursors. Due to well-known localized surface plasmon resonance (LSPR) characteristics, these NPs possess unique optoelectrical properties. NPs of the alkali and noble metals i.e. Cu, Ag and Au have a broad absorption band in the visible zone of the electromagnetic solar spectrum. Metal nanoparticles are purely made from Gold, Copper, Titanium and Silver amongst others (Khanna et al., 2019). Silver nanoparticles are extremely important due to their attractive physicochemical properties and biological functionality, including their high antimicrobial efficiency and relatively non-toxic, wide spectrum of bactericidal properties, anticancer properties and other therapeutic abilities, their unique ability to form diverse nanostructures and theirrelatively low manufacturing cost (Almatroudi, 2020).They exhibit significantly different physical and chemical properties to their larger material counterparts (high specificity and surface area). Gold, silver, copper metallic nanoparticles have been used for various medicinal and biotechnological researches.

Silver nanoparticles are one of the most synthesized nanoparticles. Silver is a gleaming, very ductile, and malleable element but slightly harder than gold, with a symbol of Ag and atomic number of 47. It is one of the basic elements that make up our planet. In nature, it exists as a native element, as an alloy combining with other metals (e.g., gold) and as minerals (e.g. hlorargyrite and argentite) (Keat et al., 2015).Chemically, silver possess four different oxidation states which are Ag^0, Ag^{1+}, Ag^{2+}, and Ag^{3+}. However, it is a chemically inactive element, but it soluble silver salts can be formed when silver reacts with nitric acid or hot concentrated sulfuric acid. It also possesses an excellent conductivity of heat and electricity, yet its applications in electrical industry have greatly been limited due to its greater cost (Galatage et al., 2021). As for metallic silver form, it is insoluble in water, but its metallic salts such as silver nitrate, $AgNO_3$, and silver chloride, AgCl, are water-soluble. Due to its good absorptivity, soluble silver compounds have the risk of causing adverse effects on health through dietary intake. silver nanoparticles (SNPs) have received the greatest attentiondue to their wide spectrum of antimicrobial activity towards Gram positive and Gram negative bacteria, fungi, and viruses (Sureshkumar et al., 2010). Moreover, SNPs have been used in various applications including dental, medical therapeutics and diagnosis, foodpackaging, catheters, textiles, and coatings (Yoksan and Chirachanchai, 2010; Lim et al., 2012). Silver nanoparticles are of particular interest in the modern research of nanotechnology due to its unique properties, which can be incorporated into a wide range of extensive applications such as antiseptic agents in medical industry, cosmetics, food packaging, bioengineering, electrochemistry, catalysis, and environmental uses. As compared to their bulk materials, noble nanoparticles exhibit different catalytic activities, and therefore nano catalysis recently has gained much attention in such a way of using nanoparticles as catalysts in various types of processes.

Owing to its multidisciplinary nature, nano biotechnology is increasingly impacting many areas of physics, chemistry, and biology. Apart from its application in drug delivery, biosensors, vaccine development, and genetic engineering, the potential role of nano biotechnology in wastewater treatment is emerging.Extracellular polysaccharides or exopolysaccharides (EPSs) are high molecular weight complex biopolymers that are majorly composed of sugars (monosaccharaides> 20) and include other non-carbohydrate organic substances, like proteins, lipids, humic substances, or extracellular DNA as their constituents (Rana and Upadhyay, 2020) which can be secreted by a microorganism into the surrounding environment. Exopolysaccharides can be synthesized to nanoparticles and these EPS biopolymers can act as both reducing and capping agents in metal nanoparticle synthesis (Garza-Cervantes et al., 2019). Having superior competence to synchronize with the metal ions, bacterial EPSs can be an effective alternative for treating wastewaters which primarily includes environmental metal wastes, pharmaceutical wastes, and wastes from the textile industry and treatment of bactericidal agents. In EPS biopolymers, reducing groups and the presence of charged functional groups which help bind the polymer with other charged moieties such as metal ions and permit effective treatment of metal contaminants in wastewater (Melo et al., 2022). Therefore, bacterial EPS can be incorporated in the synthesis of nanoparticles to reduce the toxic heavy metals in the metal-contaminated environmental wastes.

Methodology

Sample collection

Xanthan gum produced by Xanthomonas sp strain Sfl2 using sweet potato peel and characterised usingUV Vis Spectroscopy (UV-VIS), Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy Energy Dispersive X-ray (SEM-EDX) was collected from the Biotechnology Department of the Federal Institute of Industrial Research Oshodi, Lagos.

Synthesis of xanthan gum silver-mediated nanoparticles

Ten milligram of the xanthan gum was dissolved in ten millilitres of Milli Q water to from dispersion and 9Mm sliver nitrate ($AgNO_3$) was added under stirring condition. The solution was stored in a dark place at room temperature. After24 hours colour change was observed it changed to dark brown indicating the formation of polymeric silver nanoparticle. Furthermore,it was further kept for 1 month to increase the concentration of the solution. After one month the solution was centrifuged at 19200×g for 15 minutes.The pellet was collected and dried at room temperature for further analysis (Saravanan et al., 2017)

Characterisation of silver-mediated nanoparticles of xanthan gum

The synthesized silver nanoparticles were characterised using UV-Vis Spectroscopy, FTIR, SEM-EDX, Transmission Electron Microscopy (TEM) and X-ray Diffraction (XRD). This was done to reveal the shape, size, elemental composition present, functional groups present and the nature of the synthesized nanoparticles.

UV Vis Spectroscopy (UV-VIS)

EPS reduction of Ag^+ ions in aqueous solution was monitored by measuring the ultraviolet-visible absorbance spectrum of the solution the solution using a UV-VIS spectrophotometerCary 5000 UV-Vis-NIR spectrophotometer (Varian, Australia) at 300–700 nm (Rao, et al.,2016).

Fourier Transform Infrared Spectroscopy (FTIR)

Functional group analysis of xanthan gum silver-mediated nanoparticles was carried out using the method described by Kure et al., (2022).The technique involves the use of FT-IR Shimadzu 9200S spectroscope. Dried powder of silver-mediated xanthan gum was used for the analysis. The dried powder of silver-mediated xanthan gum was fused into Potassiumbromate (KBr) and pressed into pellet under pressure of the FT- IR Shimadzu 9200S spectroscope.

Scanning Electron Microscopy Energy Dispersive X-ray (SEM-EDX)

The SEM analysis was conducted to observe the micro structure and surface morphology of the xanthan gum silver mediated nanoparticles. The freeze-dried xanthan gum was fixed onto the SEM stubs and gold sputtered before observation. The SEM analysis (S-4800, Hitachi, Tokyo, Japan) was operated at 10.0 KV. The presence of elemental silver in the nanoparticles was analysed using a SEM equipped with an EDX spectrum (Hebeish et al., 2013).

Transmission Electron Microscopy (TEM)

The TEM analysis was done to observe the size, distribution and particle shape. For the analysis of TEM, a drop of SNP-containing aqueous solution was directly placed onto a carbon-coated copper grid and allowed to air dry completely prior to TEM observations. The TEM images of the nanoparticle were obtained using a **tr**ansmission electron microscope (FEI Tecnai G2 F30, Eindhoven, The Netherlands) at an accelerating voltage of 300 kV (Kanmani et al., 2013).

X-ray Diffraction (XRD)

The phase composition and crystal structure of the SNPs was determined using XRD (Philips XPERT MPD). For this, the dried sample was prepared by placing on the microscopic glass slide and the diffractogram was recorded using Cu-K _ radiation and a nickel monochromator filtering wave at a voltage and current of 40 kV and 30 mA, respectively (Kanmani et al., 2013).

Results and Discussion

Characterisation of Nanoparticles synthesized from xanthan gum was characterised using UV-Vis spectrometry, Fourier Transform Infrared Spectroscopy, Scanning Electron Microscopy Energy Dispersion X-ray, Transmission Electron microscopy, and X-ray Diffraction.

The formation of silver-mediated nanoparticles can usually be confirmed by visual observation and UV-Vis spectroscopy (Kanmani et al., 2013). UV-Vis spectroscopy analysis can provide information about the morphology, size and stabilization of silver nanoparticles. Figure 4.40 shows the UV-Vis spectra of xanthan gum silver-mediated nanoparticles after storage in the dark for 1 month. EPS reduction of $Ag+$ ions in aqueous solution was monitored by measuring the ultraviolet-visible absorbance spectrum of the solution. The xanthan gum stabilized silver nanoparticle showed a strong peak at 400–550 nm with a broad band, indicating the formation of silver nanoparticles that varied in shape and size. A distinct peak at 418.724 nm confirming the successful formation of silver nanoparticles was observed in the UV-Vis spectra. Typically, AgNP peaks in polysaccharide-based syntheses are observed between 400–450 nm as discussed in Beyene et al., (2017) and Kanmani et al., (2013). Moreover, studies by Rao et al., (2016) also confirmed the UV-Vis absorption of xanthan gum-mediated nanoparticles within this range. The intensity of the absorption increased with the concentration of silver ions during synthesis because the formation of more silver nanoparticles enhanced light absorption. Smaller nanoparticles (10–50 nm) exhibit SPR peaks around 400–420 nm while larger nanoparticles can shift the peak toward longer wavelengths. The peak's position and broadness revealed that the nanoparticles as small and uniformly distributed nanoparticles. Broader peaks suggest polydispersity or particle aggregation, while sharp peaks indicate uniform size distribution. Factors such as functional groups, concentration of silver and environmental factors can influence the absorption of light by the nanoparticle. Functional groups in xanthan gum (e.g., -OH, -COOH) can stabilize the nanoparticles and slightly shift or broaden the SPR peak due to chemical interactions and higher concentrations of silver ions or particles may lead to shifts in SPR peaks or aggregation, causing peak broadening or secondary peaks. More so, environmental factors such as pH, temperature and reducing conditions during synthesis also influence the wavelengths.

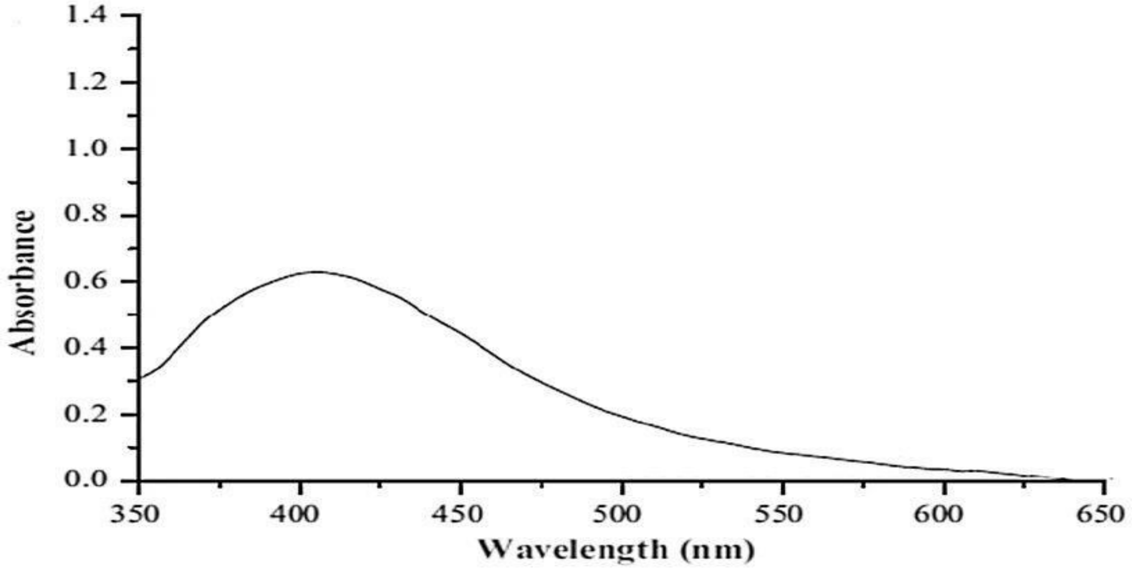


Figure 1: UV-VIS spectra of xanthan gum silver-mediated nanoparticles.

Figure 2 showed the FTIR spectra of xanthan gum silver-mediated nanoparticle. The peaks were observed ranged from 3280.1 to 1017.6 cm^{-1}. The peak located 1017.5 cm^{-1} could be attributed to C-O stretching while the peak at 1077.2 cm^{-1} revealed the presence of polysaccharide C-O-C in the silver-mediated nanoparticles and this could be as a result of the polysaccharide (xanthan gum) used for the nanoparticle synthesis. The absorption peaks at 1151 cm^{-1} and 1244 cm^{-1} indicated the presence of alkyl, amine, alkyl and ketone groups. Moreover, the peak at 1364 cm^{-1} could be attributed to C-O group while C-H bending was observed at 1420 cm^{-1}. The absorption peak at 1636.3 cm^{-1} indicated the possible presence of C=O and the peak at 2102.21 cm^{-1} revealed the presence of C=C. The C-H stretching was also observed at 2933.41 cm^{-1}.Furthermore, carboxylic acid andOH stretching of the hydroxyl group were observed at 3280.0 cm^{-1}. This was the broadest and strongest absorption peak.

Aldehyde, hydroxyl, carboxyl and esters are some of the functional groups present in the synthesized nanoparticles and the presence of these functional groups could be responsible for reduction and stabilization of silver nanoparticles (AgNp). The peaks observed in the FT-IR spectrum of xanthan gum produced by Xanthomonas sp sfl2 strain using sweet potato peel as observed by Ajayi et al., (2024) is quite different form the peaks observed in the FTIR spectrum of the xanthan gum silver-mediated nanoparticles. These observations suggest a strong interaction of Ag with the xanthan gum functional groups more so, Hydroxyl groups in polysaccharides have efficient coordination ability with Ag. Functional groups such as ester, aldehydes, hydroxyl, and carboxyl are some of the functional groups indicated in the FTIR spectrum of XGSNp and this is comparable to the works of Adebayo-Tayo and Popoola (2017); and Gomma, (2015) which also observed that silver nanoparticles synthesised from bacteria exopolysaccharides had functional groups such as ester, aldehydes, hydroxyl and carboxyl amongst others.

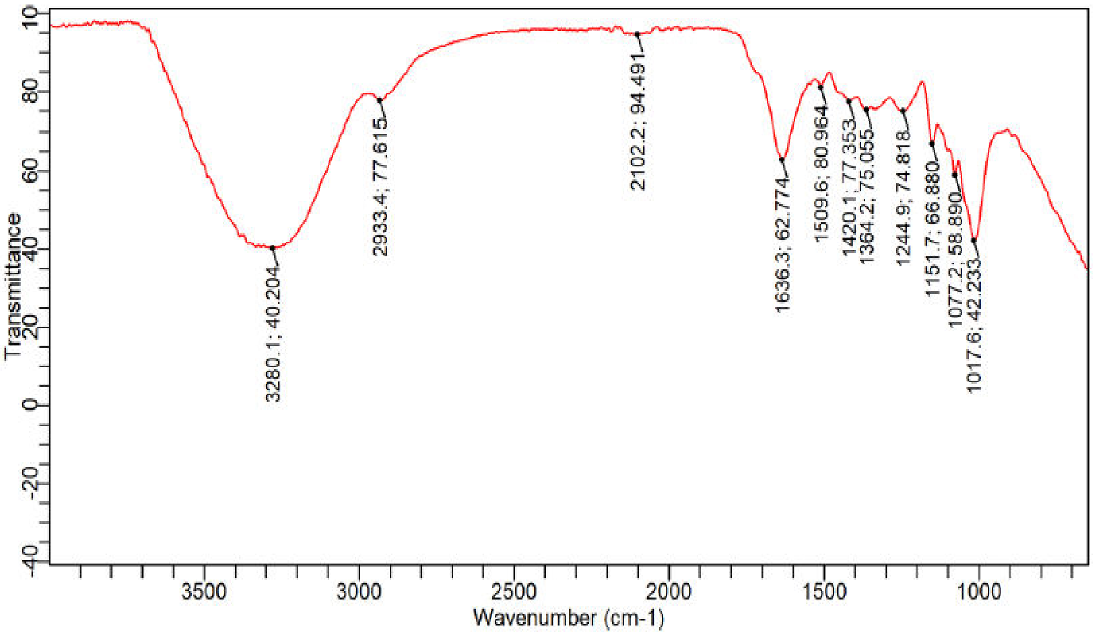


Figure 2: FTIR spectra of silver mediated xanthan gum nanoparticles.

The scanning electron microscopy revealed the shape of the silver-mediated xanthan gum nanoparticle.After synthesizing silver nanoparticles using xanthan gum derived from sweet potato peel, the SEM micrograph showed a distinct transformation in morphology. It had uniform dispersion, the xanthan gum acted as a stabilizing and capping agent, leading to a relatively uniform distribution of silver nanoparticles across the biopolymer surface. It also had a rough and granular texture compared to the plain xanthan gum, the surface becomes more granular and coarse due to the deposition of silver nanoparticles. The scanning electron microscopy revealed the shape of the silver-mediated xanthan gum nanoparticle. The SEM micrographs of xanthan gum silver-mediated at x100 (a), x1000 (b), x 4000(c), x 5000(d) are shown in figure 3 above. The xanthan gum silver-mediated nanoparticles were whitish fibre like structures which had its components aggregated.

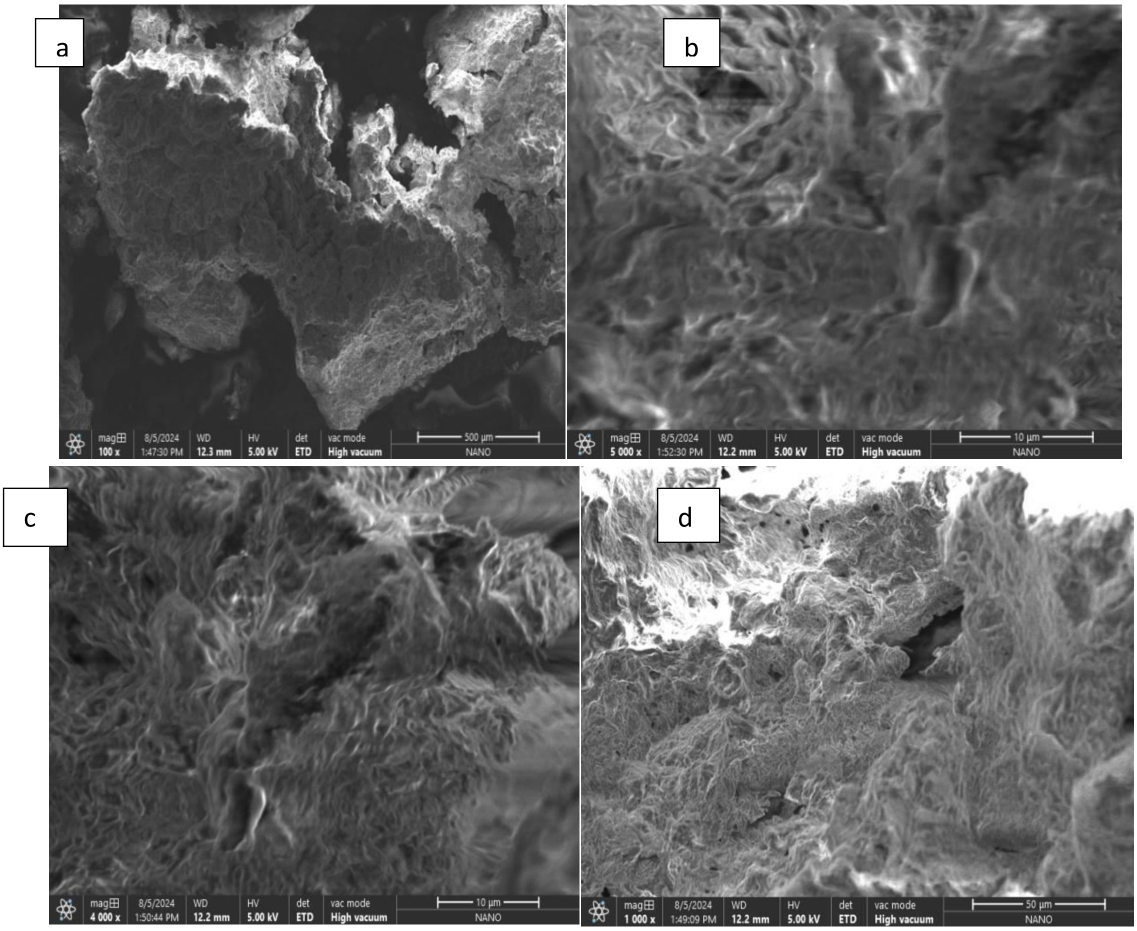


Figure 3: Scanning electron micrograph of xanthan gum silver-mediated.

The elemental analysis of xanthan gum-silver mediated nanoparticles (AgNPs) is crucial to understand the synthesis, stability, and functionality of the nanoparticles. The EDX spectrum can provide the quantitative and qualitative status of elementals in the formation of silver nanoparticles (Bindhu and Umadevi, 2013). Energy Dispersive X-ray (EDX) was carried out as shown in figure 4. The EDX profile of XGSNp showed strong signals for silver atoms (Ag). Moreover, Oxygen (O), Carbon (C), Nitrogen (N) and Vanadium (V) atoms were detected with relatively less intensity, whereas carbon this is in line with earlier works of Shukla et al., (2012) who reported that the EDX spectrum of agar-stabilized SNPs had the presence of elemental Ag in the nanoparticles. The EDX confirms the presence of silver as the primary element in the xanthan gum silver-mediated nanoparticles. Peaks corresponding to silver (Ag) validate the successful formation of silver-mediated nanoparticles while minor peaks from other elements (e.g., oxygen, carbon) are attributed to xanthan gum, which acts as a capping and stabilizing agent. The quantitative analysis provides the percentage of silver in the nanoparticles, indicating the efficiency of synthesis and reduction. Furthermore, the EDX pattern of carboxymethyl cellulose (CMC) stabilizing silver nanoparticle carried out by Hebeish et al., (2013) also revealed that the silver nanoparticle consisted of Ag, O, and C with percentages of 18.99, 47.54, and 33.47%, respectively.Atomic concentration of silver, carbon, copper, oxygen and vanadium are 82.17, 6.38, 5.95, 3.39 and 2.12 while the weight concentrations are 66.06, 19.46, 13.37, 0.64 and 0.47 for silver, carbon, copper, oxygen and vanadium respectively. Silver forms the core of the nanoparticles, responsible for their antimicrobial, catalytic, and optical properties. Silver ions (Ag^+) are reduced to metallic silver (Ag^0) by xanthan gum, which serves as areducing and stabilizing agent. The antimicrobial activity of AgNPs arises from silver's ability to disrupt microbial cell membranes, generate reactive oxygen species (ROS), and bind to cellular enzymes and DNA (Kora et al., 2021). Carbon is a fundamental element in xanthan gum, a polysaccharide composed of glucose, mannose, and glucuronic acid. Xanthan gum provides hydroxyl (-OH) and carboxyl (-COOH) groups, which act as reducing agents for Ag^+ ions and form a stabilizing layer around the nanoparticles. The carbon framework enhances nanoparticle stability, prevents agglomeration, and improves biocompatibility for biomedical uses (Beyene et al., 2017; Rao et al., 2016).

Oxygen atoms in xanthan gum's carboxyl (-COOH) and hydroxyl (-OH) groups contribute to the reduction of silver ions. These groups also assist in chelation and capping, stabilizing nanoparticles and improving their dispersion in solutions. Oxygen-containing groups enhance the binding of nanoparticles to microbial surfaces, aiding in antimicrobial efficacy (Mohana et al., 2021). Copper can act as a dopant or trace element, enhancing the antimicrobial and catalytic properties of silver nanoparticles. Copper creates a synergistic effect with silver, improving oxidative stress on microbial cells (Durán et al., 2016). Vanadium can modify the optical, electronic, or catalytic properties of silver nanoparticles. It may enhance the material's performance in catalytic reactions or energy applications, such as dye degradation or pollutant removal. Vanadium-doped nanoparticles are used in catalysis and environmental remediation (Singh et al., 2020).These elements collectively contribute to the synthesis, stability, and functionality of xanthan gum silver-mediated nanoparticles. They enhance their utility in medicine, catalysis, and environmental applications.

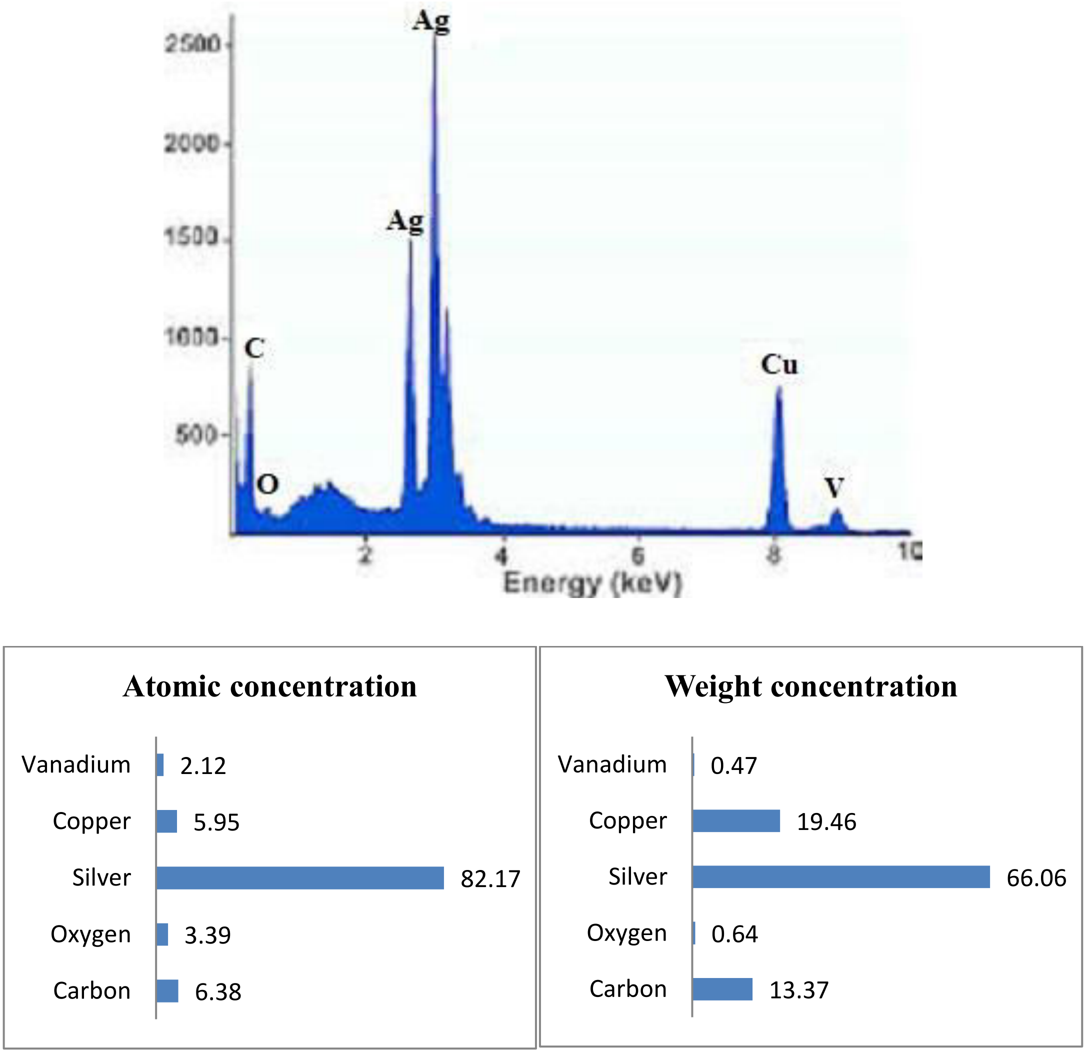


Figure 4: Energy Dispersive X-ray of xanthan gum silver-mediated nanoparticle.

The TEM micrograph revealed the size of the silver mediated xanthan gum nanoparticle ×50 nm(a) and ×100 nm(b).The size and shape of silver nanoparticle usually depend on the concentration and type of reducing and stabilizing agents used (Sharma et al., 2009). Xanthan gum a polysaccharide was used as a reducing and stabilizing agent in the synthesis of xanthan gum silver-mediated nanoparticle. The shape of the xanthan gum silver-mediated nanoparticle synthesized was spherical and the size ranged from 10.40 nm to 15.89 nm. The shape and size of the nanoparticle synthesized are similar to earlier works of Mohanty et al., 2012 and Bankura et al., 2012. Spherical shaped silver nanoparticle were obtained with an average size of 5–10 nm when silver nanoparticles were synthesized using polysaccharides such as soluble starch and dextran as reducing and stabilizing agents.

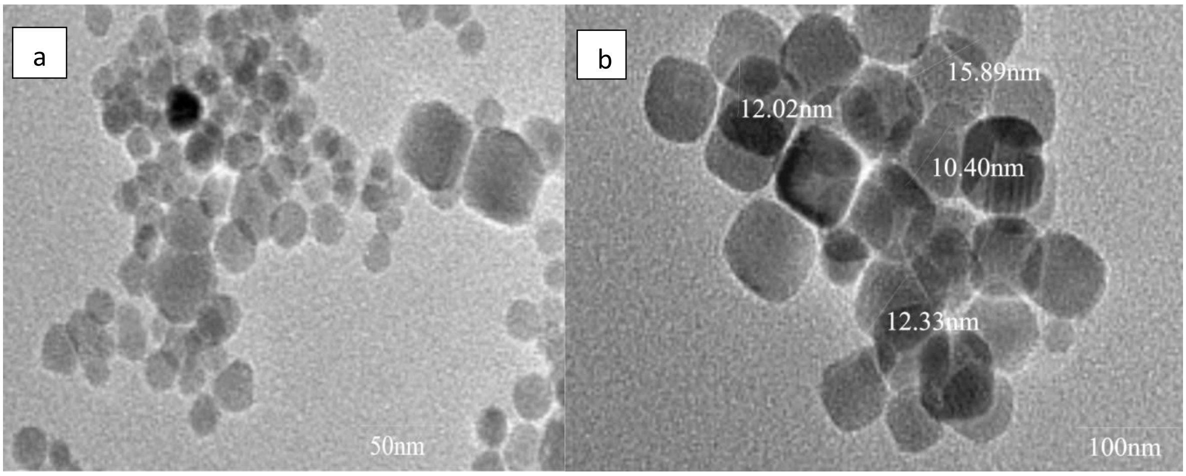
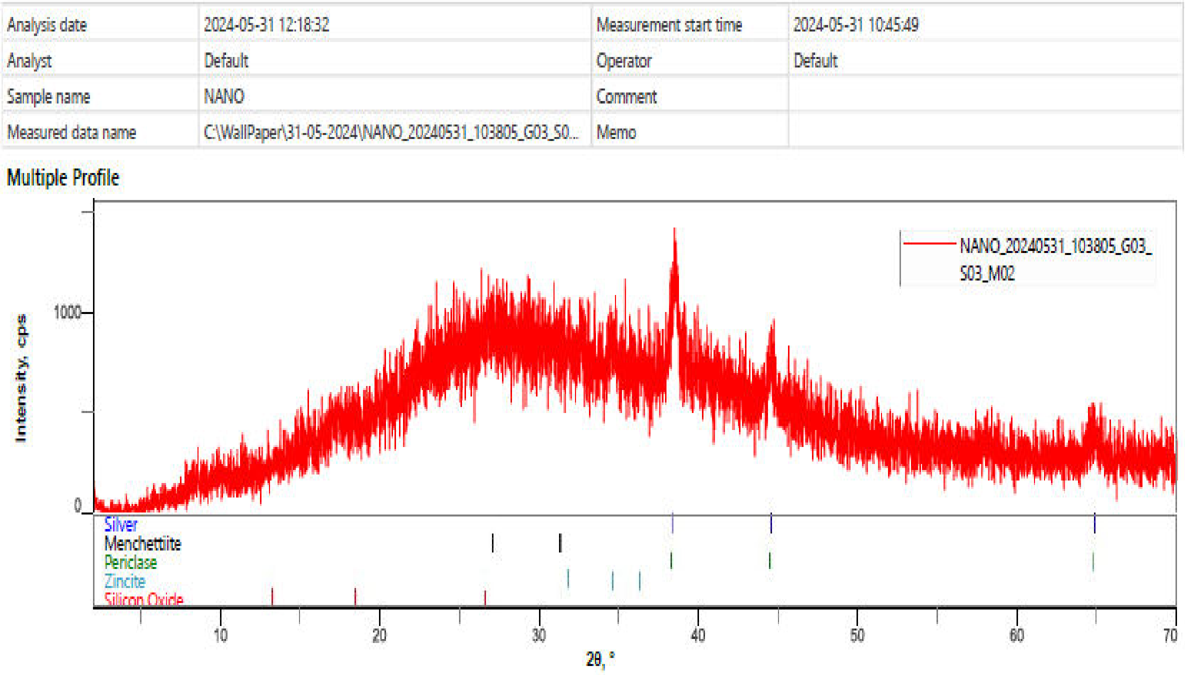


Figure 5: Transmission electron micrograph of xanthan gum silver-mediatednanoparticles.

The X-ray spectra, X-ray diffraction pattern of the xanthan gum silver-mediated nanoparticles was shown in figure 5 and 7 respectively. The X-ray diffraction (XRD) pattern of the prepared sample of silver nanoparticles was recorded and the peaks were identified by the diffractometer. Three peaks at 2θ values of 38.48, 44.47, and 64.81 degree in the experimental diffractogram have been identified to be due to silver metal and corresponding to (hkl) values - (363), (124) and (100) planes of silver and are labelled as 1-3. The XRD study confirmed that the resultant particles in the prepared sample contained silver nanoparticles. The quantification analysis using XRD revealed that the sample contained 17% of Silver (Ag). The other components showed four more peaks in the diffractogram which have been identified to be; (i) Menchettite (composed of Ag, Cu and some other metals) - 20%, (ii) Periclase (MgO) - 32%, (iii) Zincite (ZnO) - 13% and (iv) Silicon Oxide (SiO_2) - 18%. The crystalline sizes of the silver nanoparticles are observed while the full width at half maximum (FWHM) was calculated to be 0.51, 0.45, and 0.56 respectively. The value of the interplanar spacing between the atoms and lattice constant is as shown in the table and they give good information on the crystal spacing and form. Thus, the XRD analysis has shown that silver nanoparticles with well-defined dimensions could be synthesized from xanthan gum and the synthesized nanoparticles are crystalline in nature with well-arranged atoms.



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Multiple Profile

Figure 6: XRD pattern of xanthan gum silver-mediated nanoparticles.

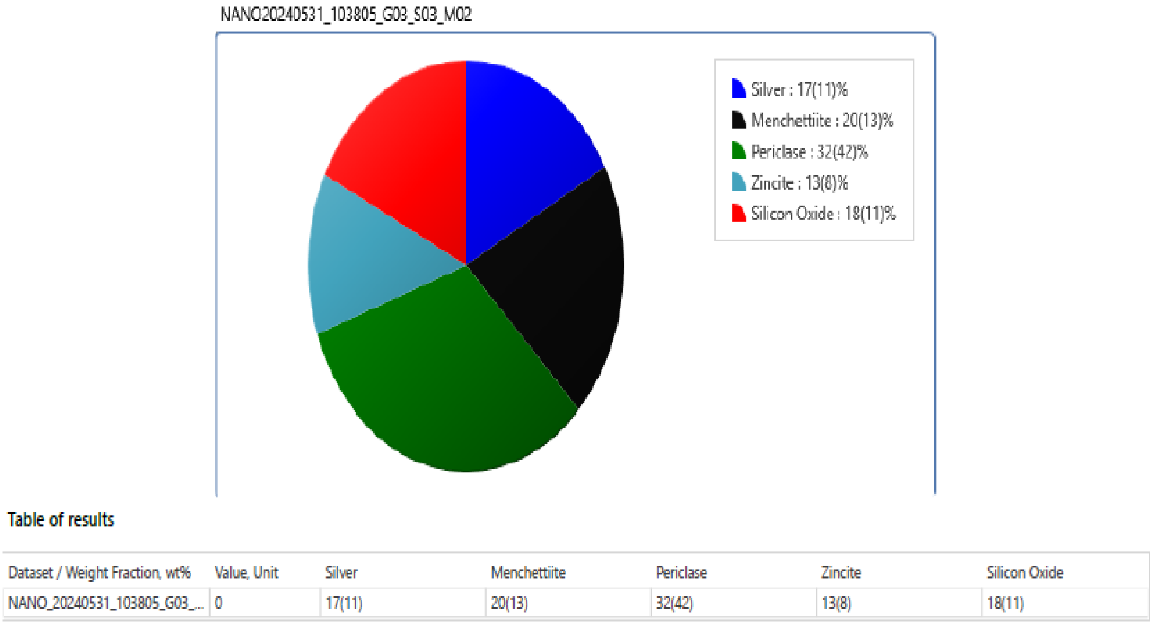

Table of results

Dataset / Weight Fraction, wt%	Value, Unit	Silver	Menchettiite	Periclase	Zincite	Silicon Oxide
NANO_20240531_103805_G03_...	0	17(11)	20(13)	32(42)	13(8)	18(11)

Figure 7: Pie chart showing the distribution of the various components of the xanthan gum silver-mediated nanoparticles.

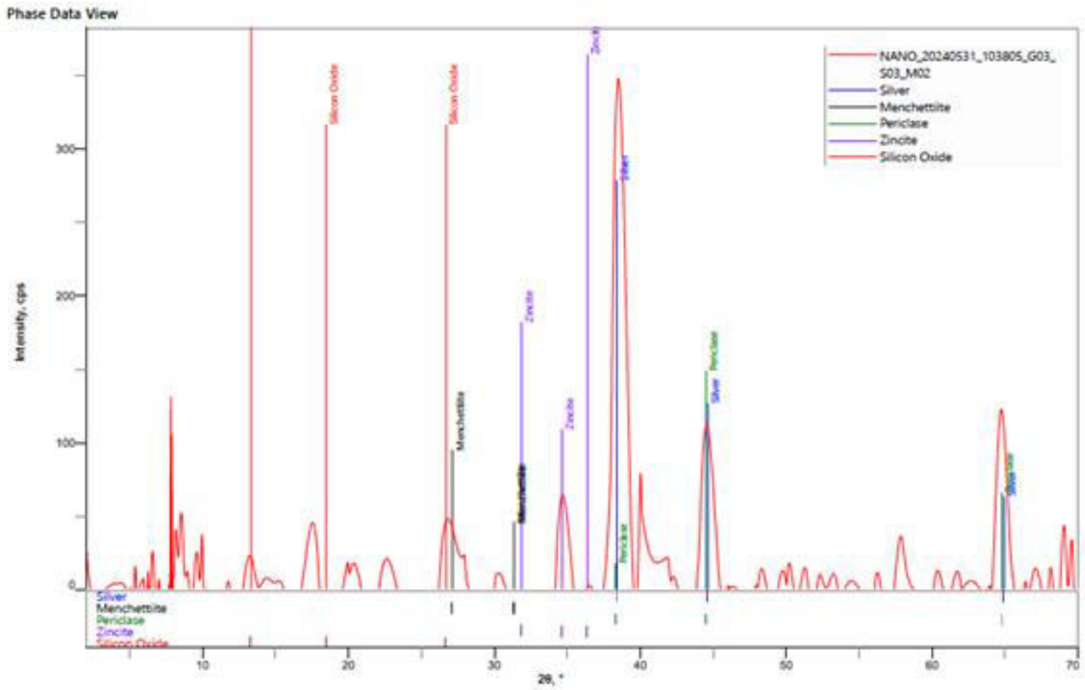

Figure 8: XRD spectra of Silver mediated xanthan gum nanoparticles.

Conclusion

Xanthan gum silver-mediated nanoparticle was successfully synthesised through green synthesis. The xanthan gum silver-mediated nanoparticles had characteristics significant for food and industrial applications and its application can be suitable due to the nontoxic nature of green synthesized nanoparticles. Further research could focus on applying the xanthan gum silver-mediated nanoparticle for water treatment (potable and wastewater), oil recovery purposes amongst others. Moreover, the toxicological studies of XGSNp are also recommended for further studies to determine its safety.

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