Poly (ethylene terephthalate) Oligomers as a Stabilizer in Colloidal Silver Nanoparticles Synthesis: Blending in Polyethylene for Antimicrobial Efficiency

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Abstract

Colloidal silver nanoparticles synthesis (CSNPs) with the chemical reduction method obtained the achievement by using PET oligomers as a stabilizer that was oligomerized from the glycolysis reaction of poly (ethylene terephthalate) (PET) and ethylene glycol (EG). The study was conducted by varying the concentration of silver nitrate ($AgNO_3$) solution, D-glucose, and stabilizer to find the most suitable conditions to control silver nanoparticles (AgNPs) size. Fourier transform infrared spectroscopy (FTIR) exposed oligomers' functional groups, the same as poly (ethylene terephthalate). The scanning electron microscope (SEM) and atomic force microscopy (AFM) revealed that the smallest size was about 42 nm for high concentration at 0.75 M of $AgNO_3$ solution, 1.00 M of D-glucose, and 1.8%w/w of the PET: EG ratio. When analyzed by an atomic absorption spectrophotometer (AAS), the AgNPs concentration was approximately 71,870 ppm. The antimicrobial efficiency test by the plate count method showed greater than 95% inhibition when using CSNPs at AgNPs concentration of 10 ppm. Furthermore, blending CSNPs in polyethylene pellets and polyethylene sheets at AgNPs concentrations of 200 ppm and 750 ppm, respectively, could provide more than 95% inhibition efficiency.

Keywords: Poly (ethylene terephthalate); Colloidal Silver Nanoparticles; Silver Nanoparticles; PET oligomers; Antimicrobial Efficiency

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1. Introduction

Colloidal silver nanoparticles (CSNPs) have received widespread attention in recent years because of their applications, especially antimicrobial and antibacterial activities [1]. Previous studies have suggested that there are several methods of preparation. However, it seems that the chemical reduction method is the most widely used because of its stability and potential in controlling silver nanoparticles (AgNPs), usually ranging from 1 to 100 nm. Generally, this method consists of reducing agents, salt solutions, and stabilizers. Initially, the reduction of salt solution with Ag^+ ions can lead to the formation of silver atoms (Ag^0) by reducing agents such as borohydride, citrate, ascorbate, alcohol, and D-glucose, where the problem of agglomeration of colloidal nanoparticles has solved by stabilizers such as polyvinyl alcohol, polyvinylpyrrolidone, and polyethylene oxide [2], [3]. Controlled synthesis of CSNPs is based on experimental conditions such as the nature of stabilizers and strong or weaker reductants that affect the particle's size, size distribution, morphology, stability, and physicochemical properties when blending in the advanced nanomaterials [4].

Blending CSNPs into polymers is an emerging area of antimicrobial materials. A. S. Maryan reported developing polyurethane (PU) properties by incorporating AgNPs into the polymer matrix to improve antimicrobial properties. The results revealed that AgNPs exhibit antimicrobial properties due to their large surface area. Other reports have published the synthesis of AgNPs-PLA nanofibers and AgNPs-cotton fabric [5]-[7]. The mechanism of antimicrobial is the inhibition of their reproducibility due to the absorption and accumulation of Ag^0 or silver ions into the cells, causing shrinkage of the cytoplasm membrane or its detachment from the cell wall, and the interaction of the silver ions with the S–H bounds of the proteins can also disrupt and inactivate cells [8]. Naturally, the collision of AgNPs in colloids tends to aggregate into larger particles, so it is necessary to have a protective layer. Various polymers can solve this problem by acting as stabilizers. Their structure influences the control over the size and morphology of nanoparticles, indicating different redox reaction mechanisms [9].

Polyethylene terephthalate (PET) is a thermoplastic polyester for use in many fields, such as packaging, medical, automotive, electronics, and garment industries. A factor associated with its widespread use, it has developed into a problem of non-biodegradable plastic waste and is a serious global environmental issue [10]. Given the negative impact of plastic waste on living organisms and ecosystems, plastic disposal and recycling have received much attention from the scientific community. The chemical recycling of PET by depolymerization can be achieved through solvolytic chain cleavage, which produces high-quality

end-products as monomers or oligomers. It is usually carried out by various methods such as glycolysis, methanolysis, hydrolysis, ammonolysis, and aminolysis at high temperatures and catalysts. According to research on the kinetics of PET glycolysis, the complete depolymerization of PET with ethylene glycol necessitates the use of a catalyst, which results in the breakdown of the ester linkage and the formation of bis(2-hydroxyethyl terephthalate) (BHET) monomer via transesterification [11],[12]. Although BHET from complete reactions cannot be achieved without a catalyst, products in the presence of oligomers have the potential to be used as stabilizers for the synthesis of CSNPs.

In this study, we used PET oligomers from the glycolysis reaction as a stabilizer for the synthesis of CSNPs. We investigated how different concentrations of silver nitrate, D-glucose solution, and stabilizer affect the size of AgNPs. Polyethylene was chosen as a polymer matrix for blending with CSNPs because of its extensive use in various fields and the possibility of creating materials with unique properties and widened applications. The uniform shape and size of AgNPs in colloids are one of the factors of our study for CSNPs synthesis because they can promote good dispersion in the polymer matrix. Finally, the feasibility of blending CSNPs into polyethylene was studied by testing the antimicrobial efficiency.

2. Research Methodology

2.1 Materials

CSNPs were prepared by a chemical reduction method using $AgNO_3$ 99.9% and D-glucose anhydrous from Ajax Finechem Pty Ltd (Australia), ethylene glycol 99.5% from Fisher Chemical (USA). The above chemicals were analytical reagent grade. The poly (ethylene terephthalate) (PET) bottle as a stabilizer was obtained by Singha packaging (Singha Corporation Company Limited, Thailand). A low-density polyethylene pellet received cooperation from InnoPlus (PTT Polyethylene Company Limited, Thailand).

2.2 Synthesis of Colloidal Silver Nanoparticles

Initially, PET oligomers were prepared by glycolysis reaction between poly (ethylene terephthalate) (PET) bottle pieces of 2 x 2 mm^2 and ethylene glycol (EG) at 0.6, 1.0, 1.4, and 1.8%w/w. The reaction temperature was about 220 – 240^oC under constant reflux and magnetic stirring for 5 h and then cooled down to room temperature. In this step, we obtained a PET oligomer solution for CSNPs synthesis. Subsequently, CSNPs were prepared by mixing 100 ml of 0.01 M silver nitrate ($AgNO_3$) solution with 0.1 M D-glucose solution, and then 150 ml of oligomer solution at different weight ratios were slowly added with an increase in temperature to 120^oC under stirring continuously for 2 h. The flow chart for the preparation of CSNPs is represented in **Fig. 1**.

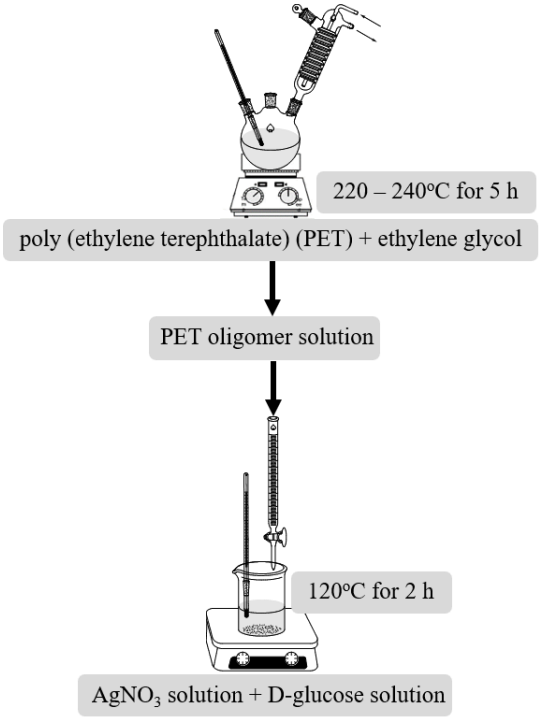


Fig. 1 The synthesis of CSNPs

After this step, we would find the suitable weight ratio of the PET oligomer solution to be used for further high-concentration synthesis. Finally, the colloidal solution with high concentrations of $AgNO_3$ solution was prepared at 0.50, 0.75, 1.00, 1.50, and 2.00 M, and varying D-glucose solution at 0.50, 1.00, and 2.00 M.

2.3 Blending and Forming of Nanocomposites

The high-concentration of CSNPs containing the smallest AgNPs were blended into polyethylene using an internal mixer (Chareon Tut MX500-D75L90) to produce 500 g of the polyethylene masterbatch at the AgNPs concentration of 5,000 ppm. The concentrations of AgNPs in polyethylene pellets were varied by varying the weight ratios of polyethylene masterbatch to polyethylene pellets at 0, 1, 2, 4, 10, and 15 phr using a single screw extruder (MASKIN TYP BX1-8). For a polyethylene sheet with a thickness of 0.3 mm, it was performed by a compression extruder (SCRIAL LP-204T) with square molding at a size of 120 mm. **Table 1** summarizes the preparation of polyethylene at various concentrations of AgNPs.

Table 1 Polyethylene preparation at various concentrations of silver nanoparticles

Sample	Concentrations of Polyethylene Masterbatch (phr)	Weight of Polyethylene (g)	Weight of Polyethylene Masterbatch (g)	Concentrations of Silver Nanoparticles (ppm)
Blank	0	500	0	0
1	1	495	5	50
2	2	490	10	100
3	4	480	20	200
4	10	450	50	500
5	15	425	75	750

2.4 Testing Antimicrobial Efficiency

The antimicrobial efficiency was determined by the plate count method. An agar plate preparation for the cultivation of microorganisms started with mixing 28 g of nutrient agar and 1,000 ml of distilled water in the beaker, and it was heated up to 260°C and kept under constant stirring at this temperature for 2 h. After that, the mixture was sterilized by autoclaving at 121°C under a pressure of 1.021 atm for 25 min, and it was cooled down to 50°C and poured into a sterile agar plate.

The testing was determined by mixing 90 ml of nutrient broth, 10 ml of microbial stock solution, and AgNPs samples in an Erlenmeyer flask and keeping them for 24 h. The sample might contain thousands, millions, or even billions of microorganisms per milliliter, so dilution was necessary for counting colonies between 30 and 300 colonies. 1 ml of sample was added to 99 ml of distilled water and mixed them together. This first dilution had a concentration (number of microorganisms per ml) of 1/100 that of the original sample. Subsequently, 1 ml of the first dilution was added to 99 ml of distilled water to make the second dilution. The second dilution had a concentration of 1/100 that of the first dilution and 1/10000 that of the original sample. This process was repeated until we had a series of dilutions.

The microbial stock solution was produced by mixing wastewater from the canal with nutrient broth and using an incubation time of 24 h. To test all samples for antimicrobial efficiency, we pipetted 0.1 ml of each dilution onto the agar plate and spread it around using a sterile glass rod, incubated at 37°C for 24 h, and then counted colonies on the surface of the agar plate. The colony counting of each sample was averaged by repeating it three times, where each dilution was based on three agar plates.

2.5 Characterization method

Fourier transform infrared spectroscopy (FT-IR, Perkin Elmer spectrum 100) analysis was performed in the range of 4000–600 cm^{-1} to determine the different functional groups present in the PET oligomer solution from the glycolysis reaction. The particle size distribution and morphology of AgNPs in the colloidal solution were assessed using a scanning electron microscope (SEM, QUANTA 450 FEI) together with atomic force microscopy (AFM, TMC Parkin-Elmer AAnalyst 800). The concentration of AgNPs was determined by an atomic absorption spectrophotometer (AAS, Varian SpectrAA 220).

3. Results and Discussion

3.1 Characterization of PET oligomers

One of the parameters for the colloidal silver nanoparticles (CSNPs) synthesis is the stabilizers. This study uses a PET bottle's pieces recycling process to obtain PET oligomers as the source of stabilizers by a glycolysis reaction between poly (ethylene terephthalate) (PET) and ethylene glycol (EG). Past research has shown that the PET chain length can be reduced by this reaction, enabling a shorter chain length

of PET of less than 130 repeating units of the polymer [13]. The complete reaction in this study is that the physical characteristics of the obtained solution are clear and colorless, which means that the digestion of the PET bottle pieces in the EG appears as a homogeneous mixture. It means that the reaction can reduce the PET chain length. The FTIR spectra of the obtained solution can be used to determine whether the functional groups differ from the PET functional groups reported in the literature [14]. **Fig. 2** shows the corresponding infrared absorption bands of the obtained solution from the glycolysis reaction.

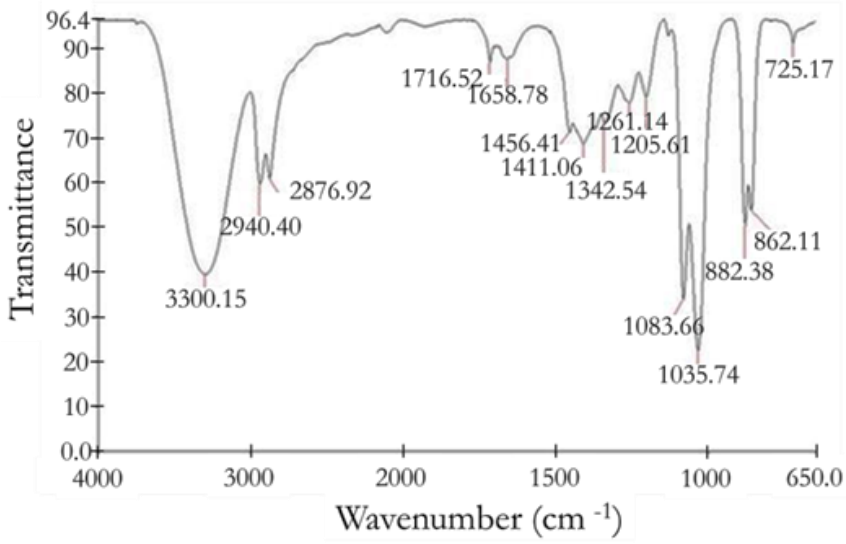


Fig. 2 FTIR spectra of the obtained solution from the glycolysis reaction

Table 2 summarizes the identified vibrational groups of the obtained solution from the glycolysis reaction. The absorption bands of the obtained solution detect the C-H bond stretching, which suggests the ethyl group and reveals the C=O bond stretching of the carboxylic acid group corresponding to the alkoxy carbonyl group. For the aromatic ring, the absorption bands can indicate the vibrations of the aromatic skeleton with stretching C=C, terephthalate group, and interaction of polar ester groups and benzene rings [14]. Based on the FTIR spectra, we believe that the product from the glycolysis reaction is only PET oligomers and does not occur in other products. Because the glycolysis reaction in our study does not use a catalyst, complete depolymerization of PET to the bis-hydroxy ethyl terephthalate (BHET) monomer cannot be achieved. These results match our requirements because PET oligomers are low molecular weight polymers with short-chain structures. They may act as better stabilizers. However, glycolysis reaction requires sufficient reaction time and temperature [15].

Table 2 Bands with assigned vibrational modes from FTIR spectra of the obtained solution from glycolysis reaction

Bands Rang (cm^{-1})	Absorption Bands (cm^{-1})	Bands
3300	3300.15	OH group (hydroxyl)
2800-3000	2940.40 and 2876.92	C-H, Symmetrical stretching
1600-1700	1716.52 and 1658.78	Stretching of C=O of the carboxylic acid group
1300-1500	1456.41, 1411.06 and 1342.54	Stretching of the C-O group deformation of the O-H group and bending and wagging vibrational modes of the ethylene glycol segment / Vibrations aromatic skeleton with stretching C=C
1100-1300	1083.66	Methylene group and vibrations of the ester C-O bond
	1261.14 and 1205.61	Terephthalate Group ($OOCC_6H_4$-COO)
800-1000	882.38 and 862.11	Aromatic rings 1,2,4,5; Tetra replaced
700	725.17	Interaction of polar ester groups and benzene rings

Fig. 3 shows the reaction mechanism between the PET chain and EG. Starting is to decompose the ester linkages in the PET chain with EG to produce oligomers, where the splitting of original molecules into two parts is the occurrence of PET oligomers. The repeating units of the original PET chain are represented by subscript m. It is always greater than subscripts n and o of PET oligomers [16].

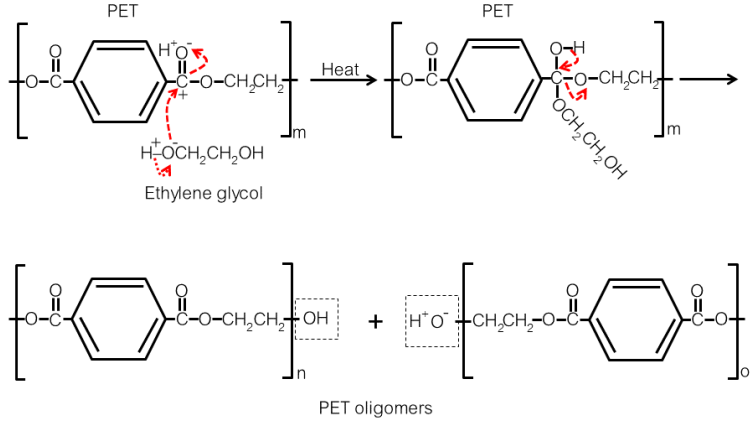
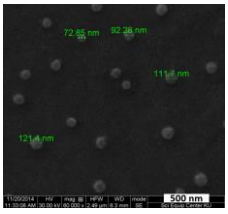
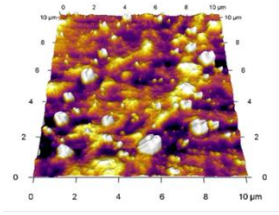
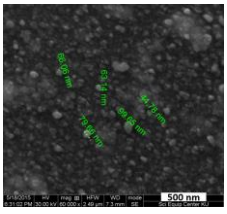
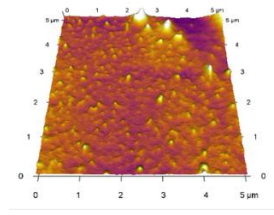
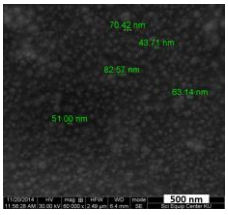
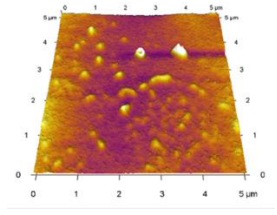
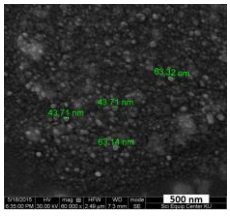
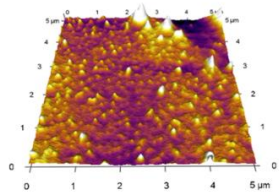


Fig. 3 The mechanism of the glycolysis reaction

We chose PET oligomers as the source of stabilizers for CSNPs synthesis at low concentrations of $AgNO_3$ solution (0.01 M) and D-glucose (0.1 M) by varying the PET: EG ratios (0.6, 1.0, 1.4, 1.8 %w/w). **Table 3** shows SEM and AFM images of AgNPs in colloid for the morphology of particles. All PET: EG ratio synthesis results in highly spherical AgNPs, and the average size of AgNPs in colloids can decrease with the increase in the PET: EG ratio. Only a few papers [17]-[20] have reported the synthesis of AgNPs with spherical shapes. The histogram for the particle size distribution for all samples is determined

to be 100 nanoparticles from SEM images. The results indicate that the condition of 1.8%w/w of PET: EG ratio for CSNPs synthesis yields a smaller mean particle diameter and a narrower size distribution; its average size is approximately 36 nm. These results are in agreement with the observed AFM images. Compared to other studies, the higher concentrations of stabilizing agents can affect the smaller size of the AgNPs. However, they also act as reducing agents, thereby accelerating the concentration of silver particles, resulting in particle agglomeration to larger sizes. Although the direct comparison of the different methods is very complex, our study has shown that the function of PET oligomers is only as a stabilizer, and the increase of PET oligomer concentration can reduce the agglomeration of AgNPs [17]. For our study, the glycolysis reaction does not use a catalyst, and the solubility between the PET bottle's pieces and EG cannot increase more than 1.8%w/w of the PET: EG ratio.

Table 3 The average size of silver nanoparticles in colloid

PET: EG Ratios (%w/w)	Average size (nm)		SEM images	AFM images
	SEM	AFM		
0	116	110-120		
0.6	63	60-70		
1.0	49	40-50		
1.4	38	35-40		

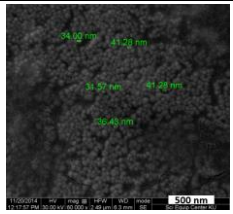
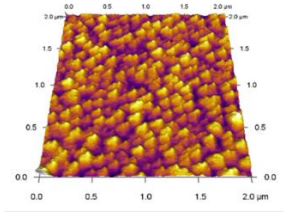
PET: EG Ratios (%w/w)	Average size (nm)		SEM images	AFM images
	SEM	AFM		
1.8	36	10-15		

Fig. 4 shows the pathway for the chemical reduction of silver ions on the PET oligomer structure. With the ability to be a reducing agent of D-glucose and EG, a plastic solvent, which is a protic solution with electron exchange capabilities. Thus, the contribution of electrons to the silver ions that enters the oxygen lone pair region of PET oligomers. A possible reaction mechanism of AgNPs formation starts with forming of silver ion-PET oligomer complexes in the carbonyl group. AgNPs are formed by deprotonating silver ions with EG and D-glucose, whereas the steric effect of a long chain of PET oligomers acts as a stabilizer of colloids which reduces the aggregation of AgNPs [9].

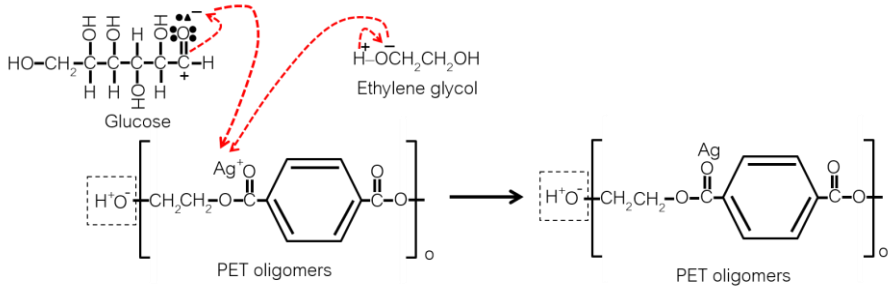
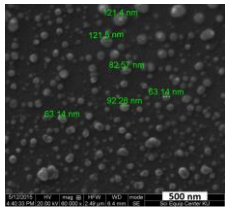
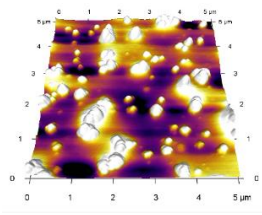
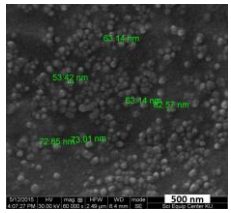
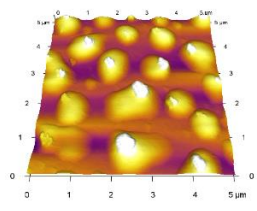
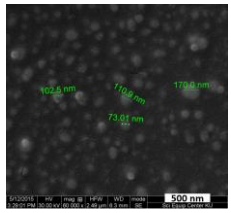
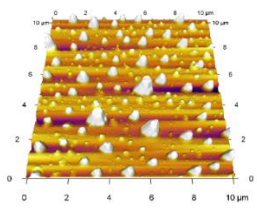


Fig. 4 The chemical reduction of silver ions on the PET oligomer structure

Our previous CSNPs synthesized data using a low concentration of $AgNO_3$ indicates that the smallest size of AgNPs can produce with the use of 1.8%w/w of PET: EG ratio. With this condition, it is possible to prepare the CSNPs using high concentrations of $AgNO_3$, which possibly reduces the amount of blending to the polyethylene matrix to avoid the change of polymer properties. 1.8%w/w of PET: EG ratio is chosen as the source of stabilizers for producing PET oligomers. D-glucose concentration is varied at 0.5, 1.0, and 2.0 M with a fixed $AgNO_3$ concentration at 1.0 M, resulting in the smallest size of the AgNPs of 58 nm with the use of a concentration of 1.0 M of D-glucose. After obtaining the suitable concentrations of D-glucose at 1.0 M, varying the concentration of $AgNO_3$ is

performed at 0.5, 0.75, 1.5, and 2.0 M. The obtained results indicate that the smallest size of AgNPs is about 42 nm with the use of a concentration of 0.75 M of $AgNO_3$, and it is also observed that they are highly spherical, well dispersed, and uniform in size. **Table 4** shows SEM and AFM images of AgNPs in colloid for the clarity of size and shape particles. The kinetics of AgNPs formation using various concentrations of $AgNO_3$ and a reducing agent is not completely surprising. It is the most popular strategy for controlling the size of AgNPs. Its concentration considerably affects the reduction rate and the nucleation and growth rates of AgNPs. In particular, in the case of reducing agents at low concentrations, partly agglomerated large particles have an appearance that has many imperfections and leads to the final precipitation. On the other hand, large clumps of AgNPs can be observed at high concentrations, which is caused by instability because the solution has high ionic strength. At the suitable concentrations, the AgNPs with well-separated exhibit narrow size distribution, and few imperfections, the size slightly decreasing with increasing concentration [18]. In this study, the calculation of the average size from histograms using SEM images is the primary data, and the supporting data is AFM images. They show the results in the same direction.

Table 4 The average size of silver nanoparticles in colloid using 1.8% w/w of PET: EG ratio

Concentration (mol/L)		**Average size (nm)**		**SEM images**	**AFM images**
$AgNO_3$	**D-Glucose**	**SEM**	**AFM**		
1.00	0.50	62	70-90		
1.00	1.00	58	60-80		
1.00	2.00	114	150-160		

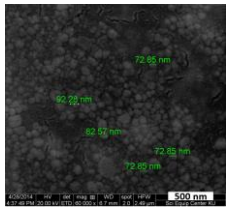
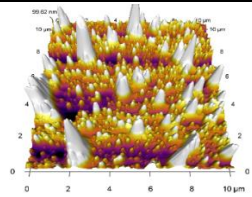
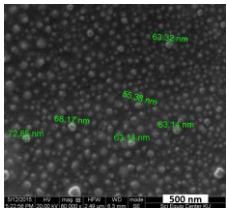
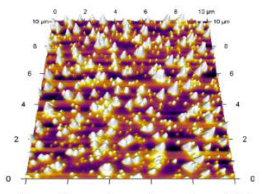
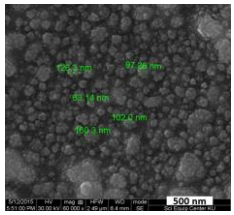
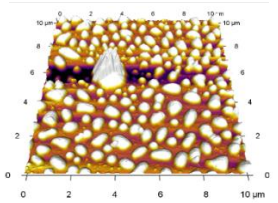
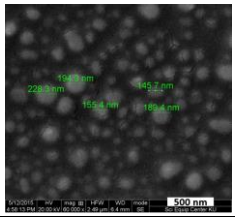
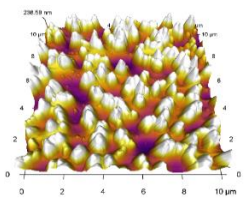
Concentration (mol/L)		Average size (nm)		SEM images	AFM images
$AgNO_3$	D-Glucose	SEM	AFM		
0.50	1.00	79	90-100		
0.75	1.00	42	40-50		
1.50	1.00	110	100-110		
2.00	1.00	183	220-240		

Table 5 shows the concentration of AgNPs in colloids using atomic absorption spectroscopy (AAS). As can be seen in 0.75 M of $AgNO_3$ solution and 1.0 M of D-glucose, the concentration after filtration is 71,870 ppm with a filtration efficiency of 98.8%, using filter paper no. 42 with a pore of 2.5 microns. Because this condition exhibits the smallest AgNPs, it reveals the highest filtration efficiency. We found that the agglomeration of larger particles tends to clump. It affects the ability to pass through the filter paper.

Table 5 Colloidal silver nanoparticles concentrations from AAS

Concentration (mol/L)		Concentrations (ppm)		Filtration efficiency (%)
$AgNO_3$	D-Glucose	Before filtration	After filtration	
0.50	1.00	49,370	41,270	83.6
0.75	1.00	72,730	71,870	98.8
1.00	1.00	91,600	88,470	96.6
1.50	1.00	157,600	128,600	81.6
2.00	1.00	191,700	106,100	55.4

Based on the initial findings of CSNPs preparation, we expected that 0.75 M of $AgNO_3$, 1.00 M of D-glucose, and 1.8%w/w of PET: EG ratio would provide the most suitable CSNPs for blending with polyethylene and produce satisfactory antibacterial activity. **Fig. 5** shows the presence of CSNPs in the polyethylene matrix with various concentrations of AgNPs in polyethylene. Because of the higher AgNPs concentration in the polyethylene, the gray color is more visible than white. This characteristic appears to be the same for the pellet or sheet.

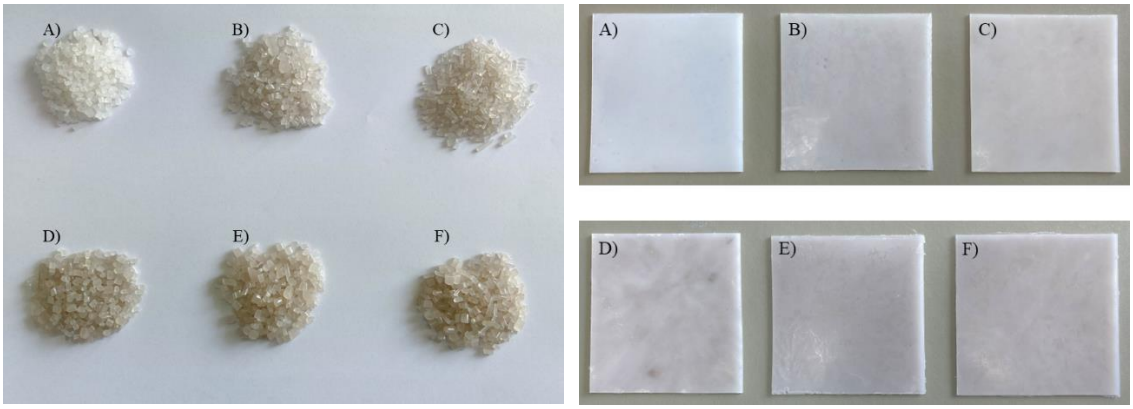


Fig. 5 The blending of CSNPs in polyethylene with various AgNPs concentrations; polyethylene pellet (left) and polyethylene sheet (right), A) = 0 ppm, B) = 50 ppm, C) = 100 ppm, D) = 200 ppm, E) = 500 ppm, F) = 750 ppm

The counting of viable microorganisms was carried out by the plate count method for testing the antimicrobial efficiency of CSNPs, polyethylene pellets, and polyethylene sheets when blended with CSNPs. After the incubation period, we can count the colonies by comparing the control sample (without AgNPs concentration) and testing sample to determine the antimicrobial efficiency (%). **Table 6** shows the results of antimicrobial efficiency testing.

Table 6 Antimicrobial efficiency of colloidal silver nanoparticles and blending in polyethylene

AgNPs Concentration (ppm)	Antimicrobial efficiency (%)		
	CSNPs	PE pellet	PE sheet
1	43.5	NA	NA
5	83.5	NA	NA
10	**98.4**	NA	NA
25	100	NA	NA
50	100	87.6	65.9
100	100	91.1	72.2
200	100	**94.6**	81.7
500	100	97.7	90.3
750	100	99.7	**96.7**

NA is meaning of “not analysis”

The antimicrobial efficiency (%) of CSNPs is 98.4% when AgNPs concentration is only 10 ppm. The inhibition of polyethylene pellets is 94.6% when blended with CSNPs at 200 ppm of AgNPs concentration. Because particles embedded within the pellet reduce the silver nanoparticle sites, higher concentrations of AgNPs are required. The blending of CSNPs into the polyethylene sheets requires AgNPs concentration of up to 750 ppm for inhibition of 96.7%. The blending of CSNPs in polyethylene requires a high AgNPs concentration to achieve the same high levels of antimicrobial efficiency. Although increasing the AgNPs concentration results in higher antimicrobial efficiency, an inhibition level greater than 90% is acceptable in most studies. Many reports have been published on the antimicrobial efficiency of AgNPs. For example, the Ag^+ concentration in silver-infused lignin nanoparticles required for high antibacterial efficiency is 20 ppm, leading to a 94% inhibition. Likewise, the use of 500 ppm of AgNPs on polyester surfaces prevents bacterial growth and biofilm formation on polyester surfaces [19],[20]. The inhibition efficiency depends on the physiological interaction between silver and microbial by involving adsorption and accumulation of microbial cells and cytoplasmic membrane contraction or detachment from the cell wall. As a result, DNA molecules condense and lose their ability to replicate when Ag ions penetrate. Silver ions also interact with the S-H boundaries of proteins, blocking and inactivating them [8]. Although the applicability of AgNPs in polyethylene in this study is less emitted when compared to lignin nanoparticles. However, the use of AgNPs concentrations in this research is similar to that of other studies.

4. Conclusion

Using PET oligomers as a stabilizer for CSNPs synthesis, the CSNPs showed colorless with varying AgNPs concentrations in the range from 41,270 to 106,100 ppm, and the sizes of the AgNPs from 36 to 183 nm. The suitable conditions can be synthesized by the AgNPs in a colloid with the smallest size of approximately 42 nm and a concentration of 71,870 ppm. In testing of antimicrobial efficiency, CSNPs showed an antimicrobial efficiency of more than 95% using the AgNPs concentration of 10 ppm, while polyethylene pellets and polyethylene sheets blended with CSNPs used the AgNPs concentrations of 200 ppm and 750 ppm, respectively. The incorporation of AgNPs into polymers can add new functionalities, making it an attractive alternative to medical or packaging applications. In particular, its use as a filler in polyethylene has attracted a lot of attention due to its wide range of applications in daily life.

5. Acknowledgement

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6. References

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