Green Synthesis of Silver Nanoparticles with Corn Straw for the Preparation of Antibacterial Paper

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A green method of synthesizing silver nanoparticles (AgNPs) with corn straw acting as reducing agents was used to prepare antibacterial paper. An ammonia solution, corn straw, and soluble starch were used as the silver precursor, reducing agent, and capping agent, respectively. The optimal condition for synthesizing AgNPs was obtained by varying the reactant ratio, temperature, and reaction time. The AgNPs were characterized by ultraviolet-visible (UV-vis) spectroscopy, X-ray diffraction (XRD), spray analyzer, and transmission electron microscopy (TEM). The obtained AgNPs were almost spherical and they were redispersed well in ethanol after centrifugation. Importantly, the prepared AgNPs were better applied in preparing antibacterial paper. After careful measurements, the mechanical properties and the antibacterial ability of the antibacterial paper were good. Therefore, the method of using corn straw as a reducing agent combined with AgNPs, to prepare antibacterial paper, is feasible. This method is noteworthy because corn straw is an underutilized material.

Keywords: AgNPs; Corn straw; Reducing agent; Antibacterial paper

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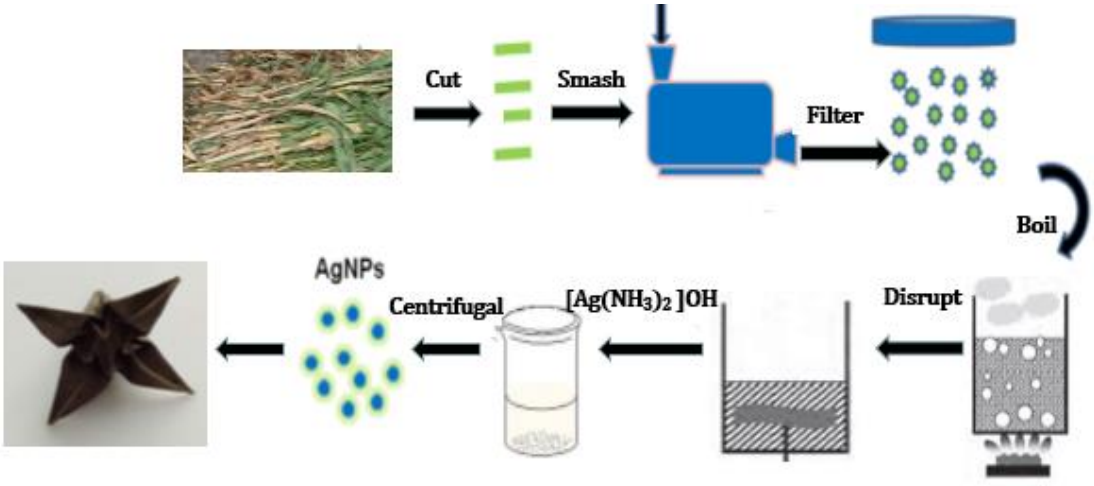
INTRODUCTION

An antibacterial agent is a functional material equipped with a bacteria killing or inhibiting ability. Dissolution-type and solution-type antibacterial agents are the main categories. The basic principle of the dissolution-type antimicrobial agents is the forming of an antibacterial ring by the antibacterial agent onto the substrate, which spreads and kills bacteria in the ring to obtain the antibacterial effect. The basic principle of the solution-type antibacterial is that the antibacterial agent on the substrate touches the bacteria directly, so it can adsorb and kill bacteria. As is well known, silver nanoparticles (AgNPs) have a strong sterilization ability, and the mechanism of AgNPs is in common with the solution-type antibacterial agents.

It can be observed that the antibacterial ability of antibacterial paper is based mainly on the quality of the antibacterial agents. There are three commonly used types of antibacterial agents: organic, inorganic, and natural. Recently, with the development of nanotechnology, AgNPs have been considered as a promising antibacterial material for preparing antibacterial paper, due to their excellent antibacterial ability (Kim and Moon 2005; Xu and Zhu 2012; Yu *et al.* 2015; Gómez-Graña *et al.* 2017). The change in the antibacterial ability of antibacterial paper frequently relates to the AgNPs and depends on many parameters, such as the size, size distribution, shape, and state of AgNPs. Due to the simple operation and their low cost, several studies based on AgNPs and antibacterial paper have been reported.

As an example of these studies, Martins *et al*. (2012) produced composites of nanofibrillated cellulose (NFC) and AgNPs *via* the electrostatic assembly of AgNP (aqueous colloids) onto NFC. The composites were applied to antimicrobial paper. Soares *et al*. (2011) reported a suitable method to produce reinforced antibacterial paper packaging using the antimicrobial triclosan (TC) and organically modified montmorillonite (OMMT) as a model group of minerals. Ling *et al*. (2013) prepared an environmentally friendly antibacterial agent, namely, AgNP-loaded quarternized carboxymethyl chitosan/organic montmorillonite (QAOM) nanocomposite, which was used to make a novel antibacterial paper by performing surface coating and internal additive methods. Nassar and Youssef (2012) constituted a recycled carton paper coated by polystyrene (PS)/silver (Ag) nanocomposites for packaging. Yu *et al*. (2014) enhanced the antibacterial activity of AgNPs/halloysite nanotubes/graphene nanocomposites through the direct growth of AgNPs on the surface of graphene-based nanosheets. However, for the AgNPs synthesis method, various toxic chemicals, such as sodium borohydride and hydrazine hydrate, are frequently applied in the process.

In previous years, growing environmental concerns have resulted in the development of new synthetic strategies avoiding the use of harmful chemicals, thus resulting in greener approaches, *i.e*. using natural products as a reductant due to their non-toxicity, low cost, and biocompatibility. Rao *et al*. (2012) rapidly synthesized AgNPs with an average size of 42 nm using an aqueous extract of tulasi leaf with $AgNO_3$ solution within 15 min. Dubey *et al*. (2010) presented a green synthesis to obtain AgNPs using the leaf extract of *Rosa rugosa*. The synthesized NPs were mostly spherical with an average size of 12 nm. Krishnaraj *et al*. (2009) used *Acalypha indica* leaf extracts to prepare AgNPs. The sizes of the AgNPs were measured at 20 nm to 30 nm. Vivek *et al*. (2012) reported a green biosynthesis of AgNPs from *Annona squamosa* leaf extract, and the biosynthesized AgNPs were predominantly spherical in shape with an average size ranging from 20 nm to 100 nm.



Scheme 1. Illustration of a high-value pathway from agriculture corn straw extraction to AgNPs for the preparation of antibacterial paper

Corn straw is the waste of corn after the corn cob is taken away. A large amount of corn straw is abandoned every year. With the development of science and technology, many methods of utilizing corn straw have been applied, while most of them are highly polluting, such as burning to produce energy. In this research, a relatively green method of

utilizing corn straw is presented. Corn straw contains a number of substances with reducing properties. Thermal cracking analysis found that the main components of corn straw are alcohols, phenol, and aromatic compounds. These components are all reducible, and this characteristic can be applied in the synthesis of AgNPs.

This study aimed to find a high-value utilization method of corn straw to synthesize AgNPs in a green way in the preparation of antibacterial paper (seen in Scheme 1). First, the corn straw extraction was obtained through a simple method. Then, well-dispersed AgNPs were synthesized based on silver ammonia solution ($[Ag(NH_3)_2]OH$) and corn straw extraction. During the synthesis, corn straw extraction was used as the reducing agent, developing a high-value pathway from agricultural residues to metal nanoparticles. Finally, the obtained AgNPs were applied in the preparation of antibacterial paper.

EXPERIMENTAL

Materials

The corn straw was purchased from local farmers (Hebei, China). Silver nitrate was supplied by Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Ammonia solution (25%) and polyvinyl alcohol (PVA) were purchased from Guanghua Sci. Tech. Co., Ltd. (Nanjing, China). Anhydrous alcohol was supplied by Chemical Reagent Co., Ltd. (Guangzhou, China). Soluble starch and anhydrous ethanol were purchased from Zhiyuan Chemical Reagent Co., Ltd. (Tianjin, China). The above reagents were of analytical purity and were used without further purification. Double distilled water was used in this experiment. Base paper was prepared manually, with a weight of 80 g/cm^3.

A JY92-IIN ultrasonic cell crusher noise isolating chamber (220V, 12MM) was purchased from Xinzhi Biotechnology Co., Ltd. (Ningbo, China).

Disposal of corn straw

First, the collected corn straw was washed with water to clean the mud. After crushing, sucrose was removed with hot water for 30 min, and disrupted with an ultrasonic cell disrupter to destroy fibroblasts, to obtain the reducing substance. After filtering, water in the extract solution was evaporated *via* freeze-drying for 8 h to obtain extract powder, and subsequently stored for the preparation of AgNPs.

Synthesis of AgNPs

An amount of 0.2 g of $AgNO_3$ was dissolved in water, a 2% $AgNO_3$ solution was obtained, and a 50-mL beaker was pretreated with diluted sodium hydroxide (NaOH, 2%). Secondly, aqueous ammonia (2%) was added into the beaker until all of the brown silver oxide was dissolved. At this point, the Ag^+ existed as $[Ag(NH_3)_2]OH$ in the clear mixture, and the $[Ag(NH_3)_2]OH$ solution was diluted to 20 mL with double distilled water. Then, 1 g of corn straw powder and 1 g of soluble starch were put into a 150-mL flask, and blended with double distilled water (18 g) for 30 min at a temperature of 80 °C. Afterwards, the $[Ag(NH_3)_2]OH$ solution was dropped into the mixed solution with a speed of 20 drops per min via a constant pressure funnel, and reacted for 30 min at a temperature of 80 °C. The mixture of corn straw and AgNPs (CS/AgNPs) was synthesized successfully under different conditions (as shown in Table 1). Next, the composite of CS/AgNPs was filtered through a 0.45-μm filter membrane to remove macromolecules. After cooling to room temperature, the composite was processed at 10,000 RPM for 10 min in a centrifuge. The

resulting supernatant was decanted, and anhydrous ethanol was added. When the dispersion was finished, CS/AgNPs solution was cleaned by an ultrasonic cleaner (Hechuang Ultrasonic Instrument Co., Ltd., Kunshan, China) for 10 min. After three washes, a small amount of AgNPs was used for characterization, and most of the product was dried at 70 °C in a vacuum drying oven. Finally, when the drying and milling had finished, the AgNPs powder was generated.

Table 1. Samples with Different Conditions of Ultrasonic Time (t_u), Reaction Time (t_r), Temperature (T), and Mixing Ratio

Samples	t_u (min)	t_r (min)	T (°C)	CS/ $AgNO_3$ (g/g)
CS/AgNPs-1	10	30	80	1.0: 0.20
CS/AgNPs-2	20	30	80	1.0: 0.20
CS/AgNPs-3	30	30	80	1.0: 0.20
CS/AgNPs-4	40	30	80	1.0: 0.20
CS/AgNPs-5	20	20	80	1.0: 0.20
CS/AgNPs-6	20	40	80	1.0: 0.20
CS/AgNPs-7	20	50	80	1.0: 0.20
CS/AgNPs-8	20	60	80	1.0: 0.20
CS/AgNPs-9	20	30	50	1.0: 0.20
CS/AgNPs-10	20	30	60	1.0: 0.20
CS/AgNPs-11	20	30	70	1.0: 0.20
CS/AgNPs-12	20	30	90	1.0: 0.20
CS/AgNPs-13	20	30	80	1.0: 0.17
CS/AgNPs-14	20	30	80	1.0: 0.23

Methods

Characterization of AgNPs

Ultraviolet-visible (UV-vis) spectra of AgNPs were collected on a spectrophotometer (UV-2201, Shimadzu, Tokyo, Japan), within a spectral scanning range of 300 nm to 600 nm under the fast-speed mode, and the scan interval was 0.5 nm. The particle size distributions of AgNPs were obtained using a Malvern laser particle size analyzer (0501601j, Malvern, Marvin, England), and an ultrasonic treatment was required before measuring. The shape and distribution of AgNPs were observed by transmission electron microscopy (TEM; JEM-2100, Japan Electronics Co., Ltd., Tokyo, Japan), which worked with an accelerating voltage of 200 kV; a few drops of the suspended AgNPs/anhydrous alcohol were placed on a copper grid coated with an ultrathin carbon film. The crystal structure of AgNPs was investigated using a D8 Advance X-ray diffractometer (XRD, Bruker, Billerica, Germany). The surface morphology of antibacterial paper observed by scanning electron microscopy (LSM710, ZEISS, Germany). The bacteria counts was observed by microscope (VMB2100DF, MicroDemo, China).

The preparation of antibacterial paper

Three different amounts of PVA powder were dissolved with double distilled water to prepare 0 wt.%, 5 wt.%, 10 wt.%, and 15 wt.% polyvinyl alcohol solutions under high temperature. The prepared AgNPs powder was evenly mixed with water to form 0 wt.%, 10 wt.%, 30 wt.%, 50 wt.%, 70 wt.%, and 90 wt.% AgNPs paste. The experiment groups are shown in Table 2. Three grams of PVA solution was mixed with 7 g of AgNPs paste to configure the antibacterial agent equipped with a different viscosity and different ratio.

Next, the antibacterial agent was evenly coated on base paper using a 10-μm four-sided finisher. After drying in a hot air oven, the antibacterial paper was obtained.

Table 2. Antibacterial Paper Coating with Different Antibacterial Agents, with Different Ratios of PVA and AgNPs

Experiment groups	PVA (wt.%)	AgNPs (wt.%)
A1	0	0, 10, 30, 50, 70, 90
A2	5	0, 10, 30, 50, 70, 90
A3	10	0, 10, 30, 50, 70, 90
A4	15	0, 10, 30, 50, 70, 90

The antibacterial activity measurements of antibacterial paper

In this study, *Escherichia coli* (*E. coli*) and *Staphylococcus aureus* (*S. aureus*) as common strains were used, and were cultured using liquid medium (Zhang and Zhou 1996; PHN 2015). The antibacterial activity in this test was estimated by determining the bacteria counts in the coated papers, as in Eq. 1,

$$X_4\ (\%) = (A - B) / A \times 100 \quad (1)$$

where X_4 is the antibacterial activity (%), A is the average colony counts of ordinary coated paper (pcs), and B is the average colony counts of antibacterial paper (pcs).

First, antibacterial papers were cut to round with a diameter of 80 mm. Then, round paper was put in the Petri dishes with corresponding size. Next, 30 bacteria counts was put in the paper separately. After 3 min, the bacteria counts were observed by microscope.

RESULTS AND DISCUSSION

Synthesis of AgNPs

The half-height and peak intensities of metal nanoparticles are closely related to particle size, size distribution, and metal nanoparticle concentration (Sun and Xia 2002). In general, the absorption peak moved to the left of the standard absorption peak (indicating a shorter wavelength) if the size of the metal nanoparticles was smaller. The greater the width of the absorption peak, the wider the particle size distribution (Sun and Xia 2003; Want 2006). Figure 1a illustrates the UV-vis spectra of obtained AgNPs with different ultrasonic times, from 10 min to 40 min. A strong absorption peak in the spectrum was centered at 410 nm, which revealed the presence of AgNPs. In this study, UV-vis spectroscopy was used to analyze the UV-visible absorption spectra of AgNPs (Voicescu *et al.* 2017). The absorption peak half width was greater with more extensive particle size distribution. Thus, 20 min was considered to be the optimum ultrasonic time in this experiment.

Figure 1b depicts the UV-vis spectra of AgNPs with different reaction times from 20 min to 60 min. As shown in the UV-vis spectra, the half-height of AgNPs gradually widened with the increase of reaction time, *i.e.* the obtained AgNPs began to aggregate due to the redundant energy, which made particle collisions possible (Baalousha 2008). Therefore, 20 min was chosen as the optimum reaction time in this experiment.

Figure 1c shows the UV-vis spectra of AgNPs with different reaction temperatures from 50 °C to 90 °C. When the temperature was 50 °C, the $[Ag(NH_3)_2]OH$ was reduced less.

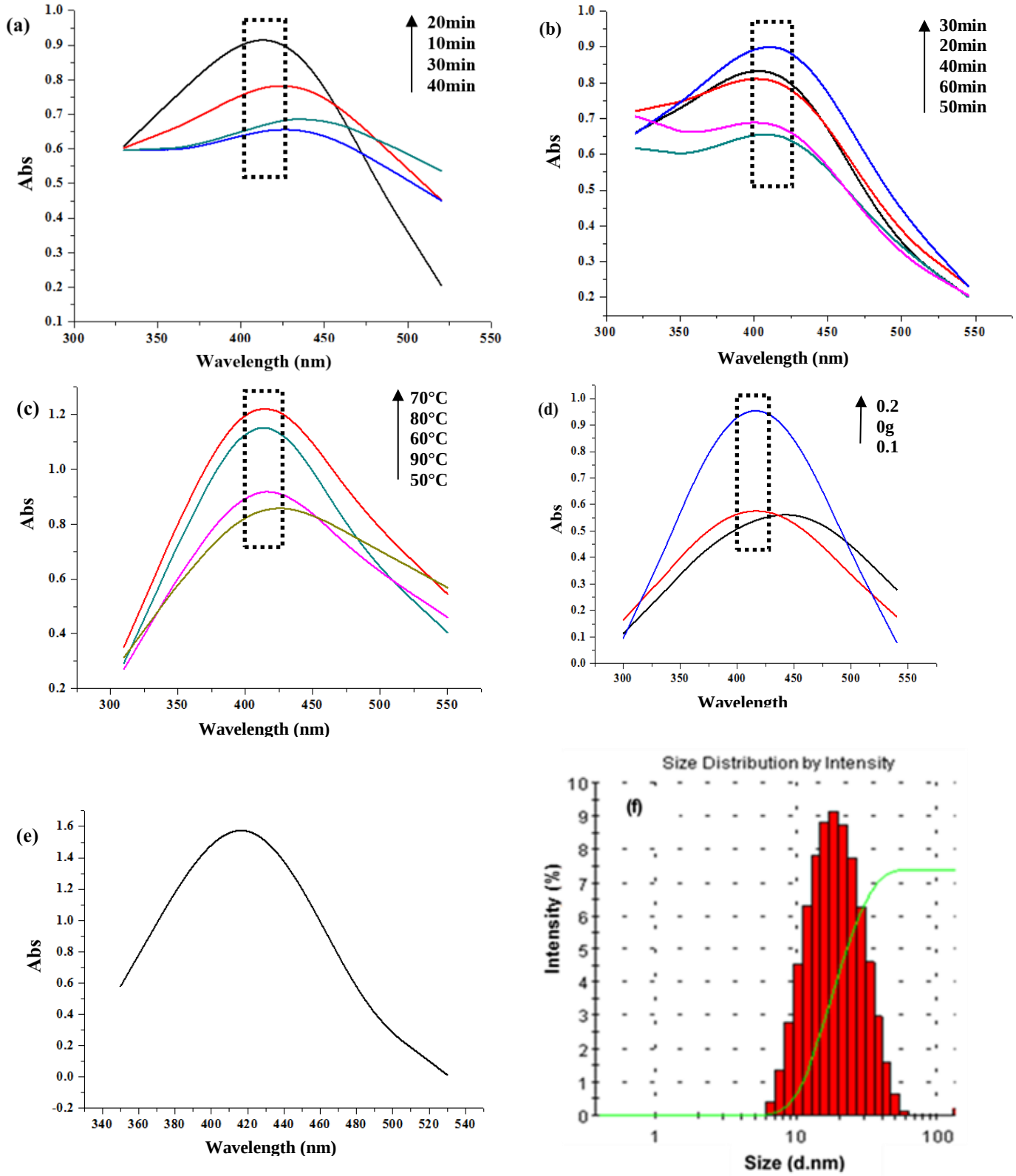


Fig. 1. The UV-vis pattern of AgNPs with (a) different ultrasonic times, from 10 min to 40 min; (b) different reaction times, from 20 min to 60 min; (c) different reacting time temperatures, from 50 °C to 90 °C; (d) different quantities of $AgNO_3$; (e) the optimum conduction: ultrasonic time of 20 min, reacting time of 30 min, temperature of 80 °C, and 0.20 g $AgNO_3$; and (f) the averaged hydrodynamic diameter distribution of the prepared nanoparticles under optimum conduction

With the increase of temperature, the absorption peak half width of AgNPs narrowed slightly, which was observed form the UV-vis spectrum. When the temperature rose to 90 °C, the absorption peak half width was greater than 80 °C. There are some reasons behind this phenomenon, such as rapid reaction speed. Considering the peak intensity and the absorption peak half width of AgNPs, 80 °C was considered to be the optimal temperature in this experiment.

Figure 1d describes the UV-vis spectra of synthetic AgNPs with different quantities of $AgNO_3$. For 0.23 g of AgNPs, a strong SPR in the spectrum was centered at 450 nm, indicating the amount of AgNPs was excessive [Shen *et al.* 2016]. The absorbance of 0.2 g was higher than that of 0.23 g, which demonstrated that the particle number of 0.20 g was more than that of 0.23 g. Therefore, 0.20 g of AgNPs was considered to be the best choice.

In summary, the optimum conditions of this experiment included an ultrasonic time of 20 min, reacting time of 30 min, temperature of 80 °C, and 0.20 g of $AgNO_3$. The corresponding UV-vis spectrum and the averaged hydrodynamic diameter distribution of the prepared nanoparticles are shown in Figs. 1e and 1f, respectively. The sharp, confined, and symmetrical absorption peaks of obtained AgNPs were narrow, and the particles were mainly spherical and monodispersed. The averaged hydrodynamic diameter distribution under the optimum condition demonstrated the particle size existed in a narrow distribution, ranging from 10 nm to 23 nm.

Structure and Morphology of Obtained AgNPs

Figures 3a, 3b, and 3d are the TEM images of prepared AgNPs, which show the shape and size of AgNPs. According to the images, the particles were almost spherical, the size was approximately 20 nm, and AgNPs was dispersed well. The AgNPs with high uniformity were favorable for mutual contact between the particles and needed to improve the antibacterial activity of antibacterial paper.

Figure 2d illustrates the XRD pattern of AgNPs. The five peaks at 38.09 °C, 44.13 °C, 64.76 °C, 77.34 °C, and 81.51 °C were assigned to diffraction from the (111), (200), (220), (311), and (222) planes of face centered cubic (FCC) silver, which was in good agreement with reference to the unit cell of the FCC structure. The peak intensity of the (111) planes was very high due to the preferential adsorption of the Ag atom on that plane during the growth process. Moreover, there were no impurities evident, which indicated that the nanosilver generated by this method had a cubic crystal structure (Xu and Zhang 2002; Li *et al.* 2007).

Surface Topography of Antibacterial Paper

Figure 3 shows the surface morphology of antibacterial paper observed by scanning electron microscopy. It can be seen that the surface of the obtained antibacterial paper is smooth.

Antibacterial Activity of Antibacterial Paper

Figures 4a and 4b show the changes in the antibacterial effectiveness of the antibacterial paper equipped with different formulations on different strains. As shown in the picture, with an increase in the content of AgNPs, the antibacterial activity of the paper against *E. coli* and *S. aureus* increased, and the antibacterial ability of the former was relatively stronger. Furthermore, the antibacterial activity of the coatings with different ratios of PVA and AgNPs were different.

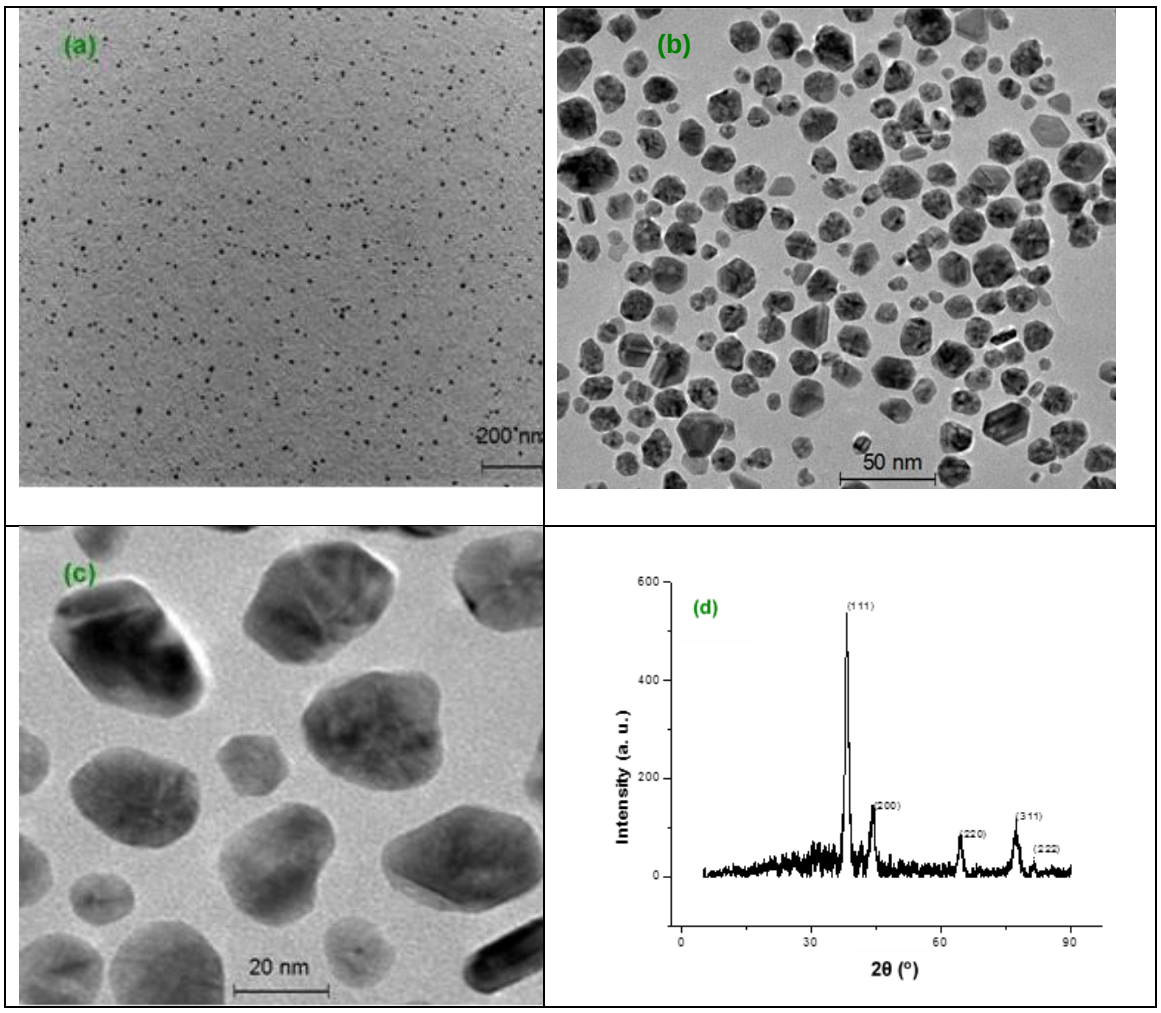


Fig. 2. (a), (b), and (c) TEM images of prepared AgNPs at different magnifications; (d) XRD pattern of AgNPs

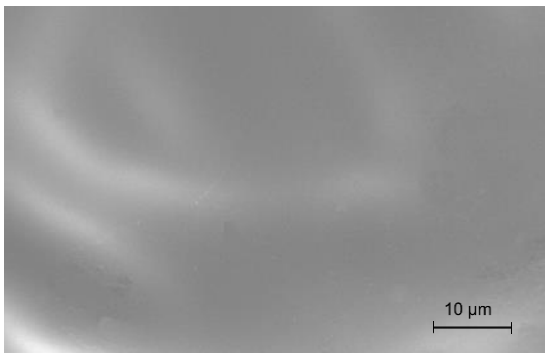


Fig. 3. The surface SEM image of antibacterial paper

The antibacterial activity was positively correlated with the mass fraction of AgNPs, and the antibacterial rate increased rapidly within the range from 0 wt.% to 10 wt.% of the mass fraction of AgNPs; while there was a relatively positive linear positive correlation after 10 wt.%. The antibacterial activity that reached the highest level as the antibacterial rate was higher than 90% when the coating consisted of 90 wt.% AgNPs and

10 wt.% PVA. Therefore, the antibacterial activity of the antibacterial paper equipped with 90 wt.% AgNPs and 10 wt.% PVA was strong.

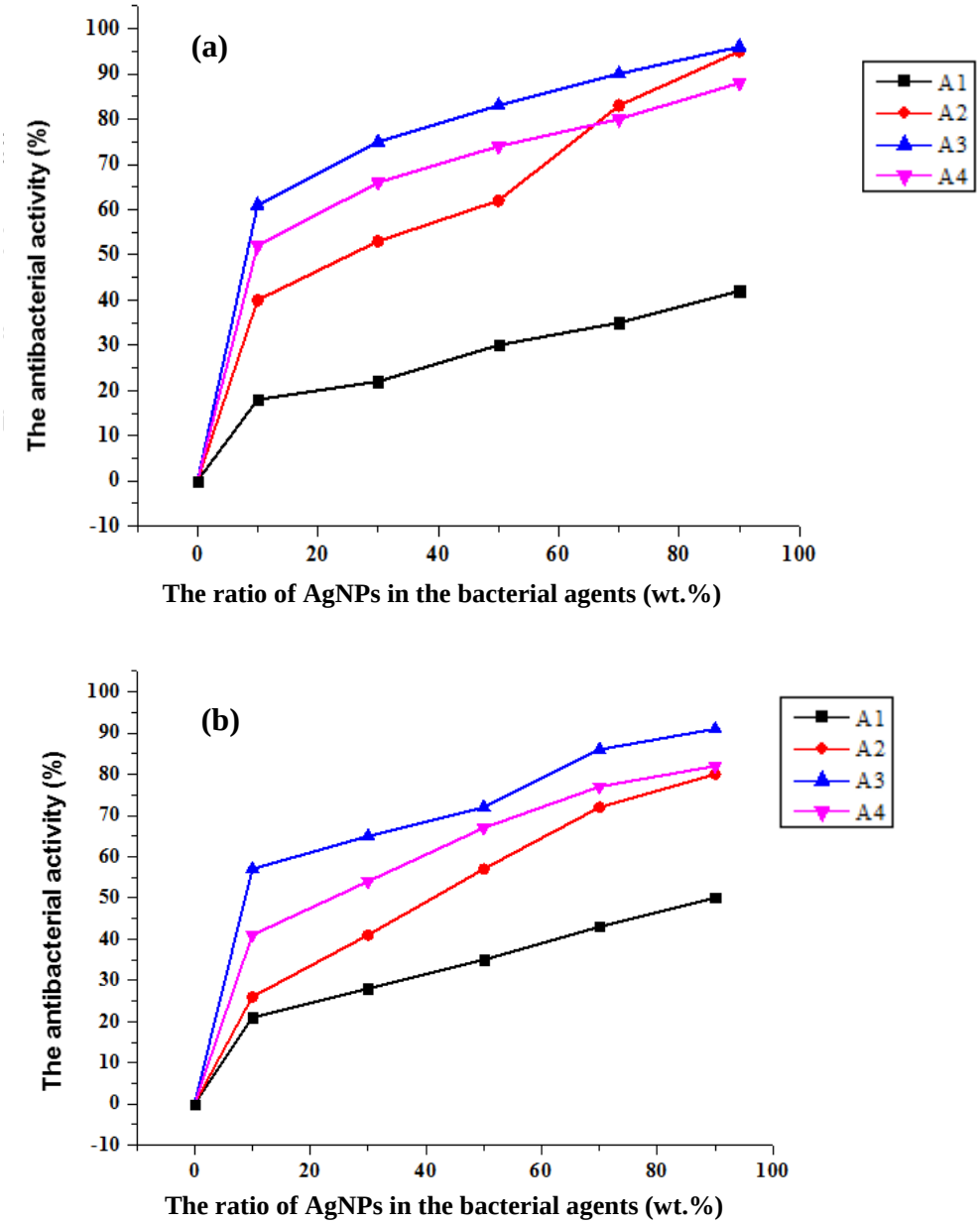


Fig. 4. The antibacterial activity of antibacterial papers equipped with different formulations on different strains; (a) *Escherichia coli*; (b) *Staphylococcus aureus*

Mechanical Properties of the Obtained Antibacterial Paper

Table 3 shows the mechanical properties of the antibacterial paper prepared starting with A3 copy paper and single-coated kraft paper (Kang *et al.* 2016). Kraft paper is a kind of paper that is commonly used in packaging applications. As shown, the bursting strength, folding times, tearing strength, and tensile strength of the antibacterial paper was good. Therefore, the obtained antibacterial paper can be applied well in the packaging field. Based on the above analysis, as well as the study of antibacterial activity, the ratio of the antibacterial agent that consisted of 90 wt.% AgNPs and 10 wt.% PVA was optimum.

Table 3. Bursting Strength, Folding Times, Tearing Strength, and Tensile Strength of Antibacterial Paper Treated with 90 wt.% AgNPs and 10 wt.% PVA

Mechanical Properties	Bursting Strength	Folding Times	Tearing Strength
Unit	KPa	HZ	mN
Antibacterial paper	412	820	735
Single-coated kraft paper	508.6	390	707.2

CONCLUSIONS

1. This study provided not only a direction for the valuable utilization of corn straw but also a novel method for the synthesis of AgNPs, which can be applied in the preparation of antibacterial paper.
2. Well-dispersed AgNPs were synthesized with corn straw as the reducing agent, and different reaction conditions were considered. The results revealed that the ultrasonication time and quantities of $AgNO_3$ had a great influence on the size distribution of $AgNO_3$, whereas the effects of temperature and time were low.
3. A high-value utilization pathway of corn straw to synthesize AgNPs for antibacterial paper was demonstrated. The obtained AgNPs were spherical and redispersed well in anhydrous ethanol after centrifugation.
4. The obtained AgNPs showed excellent antibacterial activity to *E. coli* and *S. aureus* and the antibacterial rate reached 90%. Furthermore, the mechanical properties of the prepared antibacterial paper were good, which showed a board development prospect in the preparations of wet wipes, antibacterial medical paper, antibacterial wallpaper, antimicrobial agricultural paper, *etc*.

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