

In-situ, facile and green preparation of nanoscale silver supported on activated carbon: Disinfection properties and removal of inorganic DBPs from drinking water

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ABSTRACT

Activated carbon (AC) was prepared from date palm leaves by phosphoric acid activation. The factors affecting the carbon percentage yield such as phosphoric acid concentration, synthesis temperature, and heating time were studied. The prepared AC was studied by chemical analysis of the functional groups on its surface. The prepared AC was decorated with silver nanoparticles (Ag NPs) by in-situ reduction of adsorbed silver ion. Silver ions were introduced on carbon surface by adsorption and ion exchange. Characterization of the prepared AC was performed using scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), infra-red spectroscopy (IR), and thermogravimetric analysis (TGA). Results showed that silver ions are impregnated and reduced to Ag⁰ NPs with sizes between 30 and 50 nm on the surface of AC. The disinfection properties of Ag-loaded AC were tested for Escherichia Coli and Staphylococci bacteria. The removal of some inorganic disinfection by products (DBPs) including iodate, chlorate, chlorite, and bromate were tested. The results showed that the prepared AC-Ag is effective for antibacterial disinfection and inorganic anions removal applications.

1. Introduction

Removal of bacteria from drinking water is an important process for water purification (Li et al., 2008; Pinto et al., 2020). Traditional chemical disinfection methods are widely used in the water industry but many of them produce carcinogens (Li and Mitch, 2018; Dong et al., 2019; Wang et al., 2019; Egwari et al., 2020). Recently nanoscience and nanotechnology have been applied for drinking water treatment. Nanoparticles (NPs) such as Ag, Cu, ZnO, and TiO₂, exhibit high toxicity to a number of pathogens and have been used successfully as antibacterial agent (Ruparelia et al., 2008; Zhang and Chen, 2009; Dastjerdi and Montazer, 2010; Andrade et al., 2015).

Silver nanoparticles offer excellent antibacterial action and low

toxicity to human cells which make them among the most applicable in the disinfection field (Qing et al., 2018; Bruna et al., 2021). However, some important issues require resolution prior to their industrial application such as their sustainability, retention in the water treatment system, dispersion, and cost (Ding et al., 2019; Bruna et al., 2021). According to Al-Issai et al. (2019), the nanoparticles used for disinfection should not aggregate in water, collection of the nanoparticles after treatment must be possible, and the antibacterial activity of the nanoparticles should be continuously strong.

Silver nanoparticles were impregnated previously on many supporting materials and used for disinfection purposes (Choi et al., 2008), where they were supported on carbon-based materials (graphene oxide) (Bao et al., 2011; Das et al., 2011; Zhang et al., 2011), multi-walled

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carbon nanotubes (Li et al., 2013), activated carbon (AC) fibres (Park and Jang, 2003), plasma AC (Srinivasan et al., 2013), Iron oxide, alumina, titanium oxide (Goei and Lim, 2014; Tartanson et al., 2014), and polyvinyl chitosan hydrogel (Nguyen and Liu, 2014). Also, silver nanoparticles were impregnated on the surface of AC after reduction by sodium tricitrate under UV irradiation (Yan et al., 2011). Biomass wastes are used to prepare activated carbon because they are renewable sources and available at a low cost such as rice husk (Menya et al., 2018), Moringa oleifera (Santos et al., 2020), coffee (Chiang et al., 2020), tea (Tuli et al., 2020), banana corm (Marichelvam and Azhagurajan, 2018), peanut shell (Lawal et al., 2017), and pineapple plant leaves (Beltrame et al., 2018). Moreover, activated carbon was synthesized from several palm waste for various applications such as the stem and sheath fibres (Maia et al., 2021), leaves (Soliman et al., 2016), shell (Adinata et al., 2007), kernel shell (Tobi et al., 2019), seeds (Shawabkeh et al., 2014), and palm oil fronds (Salman, 2014). However, to the best of our knowledge palm leaves-AC impregnated with silver nanoparticles is not studied.

The in-situ reduction of silver nanoparticles is expected to increase their sustainability and retention on the surface of AC. In previous studies, silver nanoparticles were prepared by reduction using chemical reducing agents such as sodium borohydride and aniline, as well as green reducing agent such as glucose and pine mushroom extract then impregnated to the surface of activated carbon (Tang et al., 2013; Anthony et al., 2014; Wimmer et al., 2018). However, the retention and sustainability of the impregnated Ag-NPs was low. Therefore, research on in-situ reduction methods for making impregnated Ag-NPs on AC surface that is stable (with minimal leaching to solution) is needed. Recently, in-situ reduction of silver NPs on the surface of oxygenated mesoporous carbon fabric without using reducing agent was reported (Qian et al., 2013).

In the present study, AC surface is used as a reducing agent and a template for the prepared Ag NPs. The presence of $-COOH$ and $-OH$

result of this work can be utilized for direct implementation as a cheap product to be used for removing pathogen from water, or as a new element in existing filtration system for inactivation of microorganism which may cause biofouling in downstream filters.

2. Materials and methods

2.1. Preparation of AC from date palm leaves

The carbonaceous precursor used for preparation of AC was date palm leaves. The leaves were collected from the UAE University campus. Prior to their use, the samples were cut to small pieces and washed to remove impurities present on the surface, then dried in a drying oven for 5 h at 70 °C. The samples were then used for studying the effect of phosphoric acid concentration, carbonization temperature, and carbonization time on the percentage yield of AC.

To study the effect of carbonization temperature, 10 g of the leaves were soaked in 50% phosphoric acid for 24 h, then carbonized for 5 h at different temperatures: 200, 250, 300, 350, 400, 450, and 500 °C. The weight loss was measured, and carbon percentage yield was calculated.

To study the effect of acid concentration, 10 g of the leaves were soaked in phosphoric acid with different concentration (10, 20, 30, 40 and 50%) for 24 h at 70 °C. The samples were filtered and washed with deionized water 5 times. The samples were then carbonized at 250 °C for 5 h. The percentage yield was calculated and recorded.

The carbonization time plays an important role in the quantity and development properties of AC (Saleem et al., 2019). Therefore, 10 g samples previously soaked in 50% phosphoric acid were heated at 250 °C for different time intervals (0–5 h), and the percentage yields were measured. All carbonizations were carried out using a Muffle furnace (CARBOLITE). The carbon yield percentage was calculated as follows:

$$\text{Carbon Yield (\%)} = \text{mass of carbon produced (g)} / \text{mass of dehydrated leaves (g)} \times 100$$

quinone-like groups and lacton groups on AC surface can act as anchoring, reducing, and stabilizing agents for the silver ion. Silver ions are adsorbed on the surface of AC by ion exchange with hydrogen ion of the carboxylic group (Srinivasan et al., 2013). This is confirmed by lowering the pH of the solution. The reduced potential of Ag^+/Ag^0 is $E^0 = 0.699$ V, simultaneous oxidation potential of hydroquinone/quinone $-OH/-O$ is $E^0 = 0.9$ V. Therefore, it is possible for Ag^+ to spontaneously be reduced on the surface of AC since electrons can be donated from carbon surface which has a lower reduction potential.

The cost of AC precursor and method of treatment are mainly responsible for the cost of the final material. Therefore, adsorbents' cost is crucial for the process for designing and installing economical water treatment systems (Duan et al., 2020). By combining silver NPs with the removal potential of AC for inorganic DBPs beside the low-cost resources of AC, it is possible to create an integrated disinfection system for removal of adsorbent with improved removal potential for DBPs. To the best of our knowledge, in-situ reduction of silver ions on the surface of AC prepared from date palm leaves was not studied before.

In the present study, preparation of AC from low-cost agricultural waste (i.e. date palm leaves) is carried out by phosphoric acid activation. Furthermore, impregnation of AC with silver NPs is carried out by ion exchange followed by in-situ reduction of silver ion. In addition, the strength of the prepared Ag NPs impregnated AC for disinfecting of Escherichia Coli (E-Coli) and Staphylococci bacteria is studied. Furthermore, the ability of the prepared materials for removal of chlorate, chlorite, and bromate from drinking water was tested. The final

For neutralisation, the carbonized samples were washed by 1% $NaHCO_3$ until the pH of the solution is adjusted to 6. Then the samples were washed by deionized water to remove any residual chemical agents. Finally, the samples were dried at 70 °C for 4 h. Complete grinding of samples was carried out by mortar and pestle then sieved by mesh size between 425 and 710 μm . The samples were then used for further oxidation by nitric acid. Oxidation by high concentrated nitric acid (10 M) introduces more functional groups on the surface of AC, then washed and used for impregnation with silver (Menendez et al., 1995). This oxidation step changes the distribution of functional groups but does not alter drastically the physical properties (surface area and porosity).

2.2. Impregnation of AC with Ag NPs

2.2.1. Impregnation with Ag

The term impregnation can be defined as the fine distribution of chemicals or metal particles in the pores of AC (Cusioli et al., 2020). The introduction of metal particles to the carbon surface can be through ion exchange, excess solution impregnation, incipient-wetness impregnation, and/or physical vapour deposition method (Paredes-Doig et al., 2020). In the present study silver ions were introduced to the surface of AC by adsorption and ion exchange. In-situ reduction takes place then on the surface of AC (Jia and Demopoulos, 2003). Carboxylic, hydroxyl, and laconic groups on the surface of AC are used as anchoring and

reducing sites for Ag^+ . Anchoring is achieved by ion exchange with hydrogen ions of the carboxylic group and reduction with hydroxyl group.

Herein silver ions are sorbed on the surface of carbon from solution by ion exchange with the surface functional group of AC or by adsorption. In the current preparation method, UV-radiation was used to enhance the photo-reduction of silver ions (Yan et al., 2011), and sonication was used to enhance the size of the formed Ag NPs on the AC surface and to avoid aggregation.

Five grams of AC were equilibrated with 100 mL of 0.001 M silver nitrate, then sonicated for 3 h under UV irradiation at a wavelength of 365 nm. The mixture was shaken for 2 h at 300 rpm at 25 °C. The prepared AC-Ag was separated from solution by filtration using a Buchner funnel then washed with 0.01 M ammonia solution to remove any precipitated silver chloride and complete the reduction of adsorbed silver ions. Finally, the samples were washed with distilled water and dried at 80 °C for 4 h.

2.2.2. Point of zero charge surface functional groups determination.

The pH_{pzc} was measured by a Melvern Nanosizer system. The acidic and basic functional groups were determined by titration according to Boehm (Boehm et al., 1964).

2.3. Characterization of the prepared Ag-Loaded carbon

2.3.1. X-ray powder diffraction spectroscopy (XRD)

The X-ray diffraction analysis for the silver loaded and unloaded AC were carried out using a Shimadzu 6100 X-ray diffractometer, with $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$)

2.3.2. XPS analysis

The chemical state of elements on the AC surface was investigated using X-ray photoelectron spectroscopy (XPS) using an ESCALAB-250 system with $\text{AlK}\alpha$ X-ray.

2.3.3. Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX)

SEM and EDX analyses were carried out using a Jeol scanning electron microscope (JSM-840). Both SEM and EDX analysis were carried out for silver loaded and unloaded AC samples.

2.3.4. Thermogravimetric (TGA)

TGA analysis was carried out for AC before and after impregnation with silver using a Shimadzu TA 60 TGA analyser. 5 g of each sample were placed in a platinum crucible and heated up to 600 °C in air atmosphere with a heating rate of 15 °C/min.

2.3.5. Disinfection properties of Ag NPs

The disinfection properties of the prepared AC were tested by the inhibition zone and inactivation rate method (Srinivasan et al., 2013). For the inhibition zone determination, 0.2 g of AC and AC-Ag powder were added separately into 10 mL of water and mixed well. The holes were then filled with 20 μL of different types of AC-Ag solutions. The plates were incubated at 27 °C for 24 h. After incubation, the diameter of the clear zone of inhibition surrounding the AC was taken as a measure of the inhibitory power of the AC-Ag against tested bacterial strains (E-Coli and Staphylococci).

In the inactivation rate method, 0.2 g of AC-Ag were equilibrated separately with 100 mL of 1000 E-Coli. The beakers containing bacterial suspensions were kept on a shaker at 37 °C for 1 h and fractions were drawn after 15, 30, 45, and 60 min to test their bacterial reduction as a function of time. The membrane filter technique was used for bacteria analysis. Spiked tap water of 1000 CFU (colony-forming unit) without AC was used as a control sample.

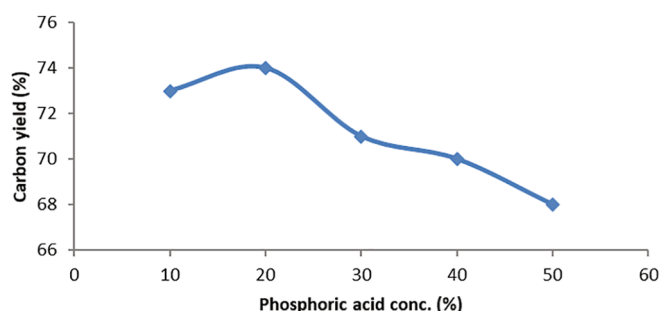


Fig. 1. Effects of phosphoric acid concentration on AC yield (Temp. 250 °C and heating time 5 h).

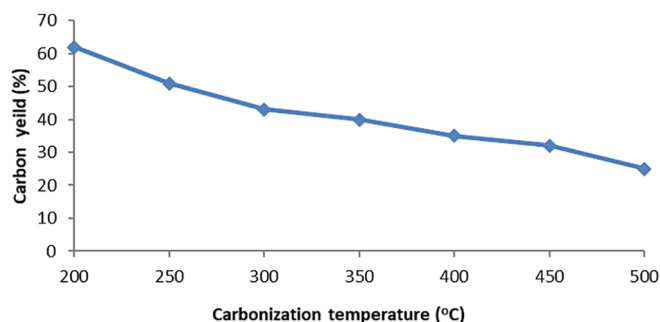


Fig. 2. Effects of heating temperature on AC yield (Acid conc. 50% and heating time 5 h).

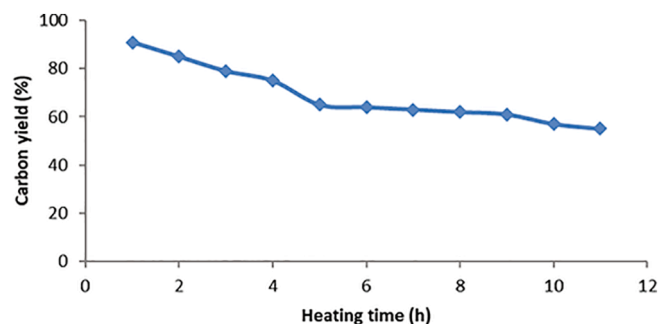


Fig. 3. Effects of heating time on AC yield (Acid conc. 50% and heating temperature 250 °C).

2.3.6. Removal of inorganic DBPs using AC-Ag

Removal of chlorate, bromate, chlorite, and iodate DBPs by the silver NPs impregnated AC was studied in a batch system. 25 mL of different concentration (10, 1, and 0.1 mg/L) of DBPs were mixed with 0.1 g of AC. The mixture was agitated at room temperature for 24 h. The mixture was separated by centrifugation at 250 rpm and filtered with a micro-pore filter (45 μm), then analysed by ion chromatography (Dionex IC-90) supplied with an AS-14A column and a conductivity detector for determination of the remaining DBPs. The removal percentage of DBPs was calculated according to:

$$\text{Removal}(\%) = \frac{(C_o - C_f)}{C_o} \times 100$$

Where C_o and C_f are the initial and final concentrations of DBPs, respectively.

The adsorption tests were carried out in batch experiments by equilibrating 0.5 g of impregnated AC with 100 mL of 10 ppm of bromate solution. The mixture was shaken at 250 rpm. Samples of 1 mL were collected at different time intervals, filtered, and analyzed for

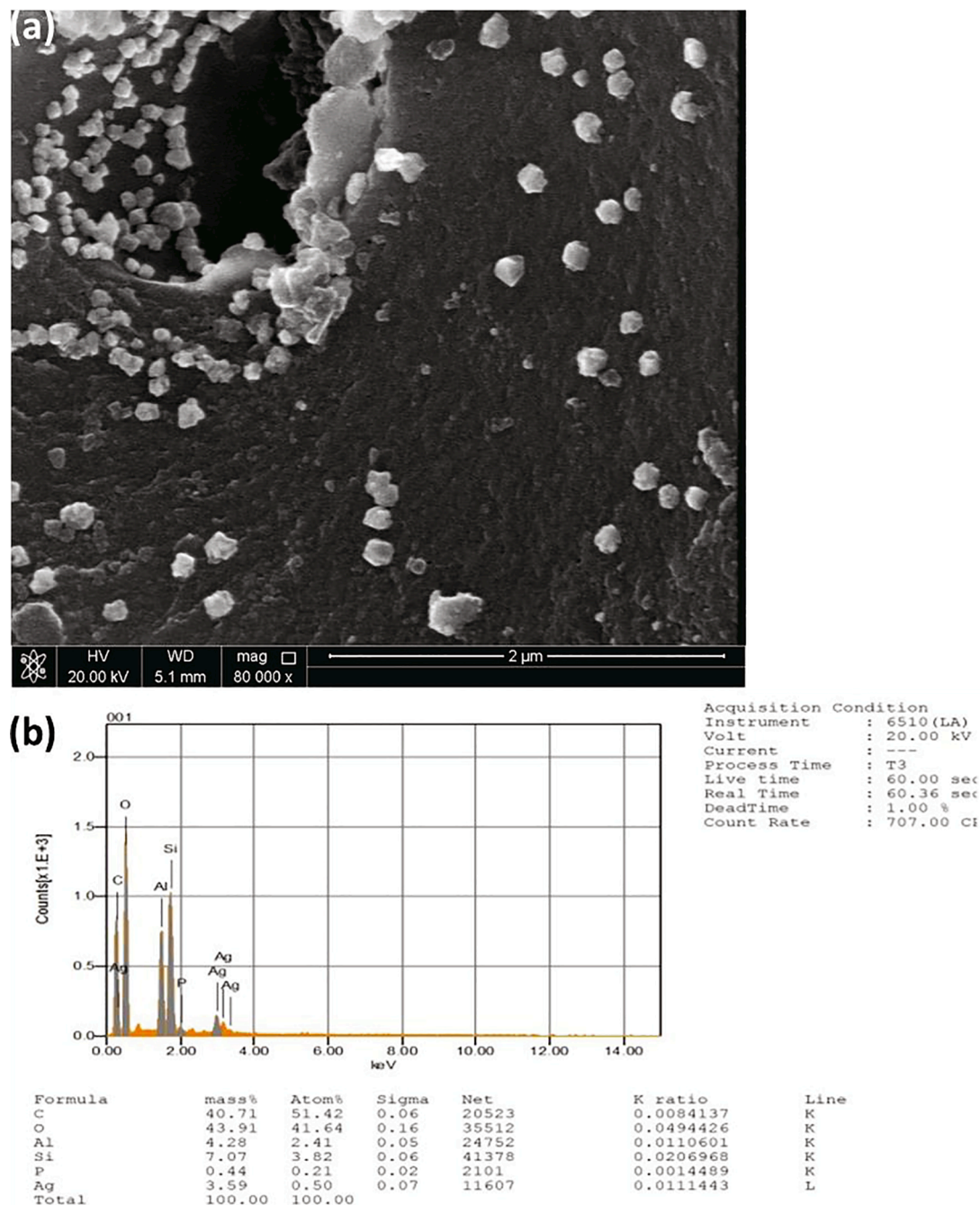


Fig. 4. SEM (a) and EDX (b) of AC impregnated with Ag.

bromate concentration.

3. Results and discussion

3.1. Preparation of AC and impregnation with Ag NPs

3.1.1. Preparation of AC

Conditions affecting the properties and percentage yield of the prepared AC such as phosphoric acid concentration, carbonization temperature, and time were investigated. Fig. 1 shows the dependence of the percentage AC yield on acid concentration. The figure reveals that the yield percentage of AC increases with the increasing concentration of phosphoric acid. The dependence of the percentage yield on carbonization temperature is shown in Fig. 2. The yield percentage of AC

decreases with increasing carbonization temperature. Similarly, the yield percentage of AC decreases with increasing carbonization time as shown in Fig. 3. Those results can be understood in terms of the increase of combustion of the material and generation of carbon dioxide with increasing carbonization temperature and time (Daud et al., 2001).

3.1.2. Impregnation of AC

Fig. 4 shows an SEM image (a) and EDX analysis (b) of the AC loaded with Ag NPs. The NPs are mainly concentrated on pore openings of the external surface. This can be attributed to the concentration of oxygen function groups on the surface of AC when it is oxidized with a strong oxidizing agent. The composition of silver NPs is confirmed by EDX analysis.

AC surface is considered a semiconductor (Velo-Gala et al., 2013)

File Name : Standard\Geo
 Sample Name : 1
 Date & Time : 07-21-14 15:05:10
 Condition
 X-ray Tube : Cu(1.54060 Å) Voltage : 40.0 kV Current : 30.0 mA
 Scan Range : 10.0000 <-> 80.0000 deg Step Size : 0.0200 deg
 Count Time : 0.30 sec Slit DS : 1.00 deg SS : 1.00 deg RS : 0.30 mm

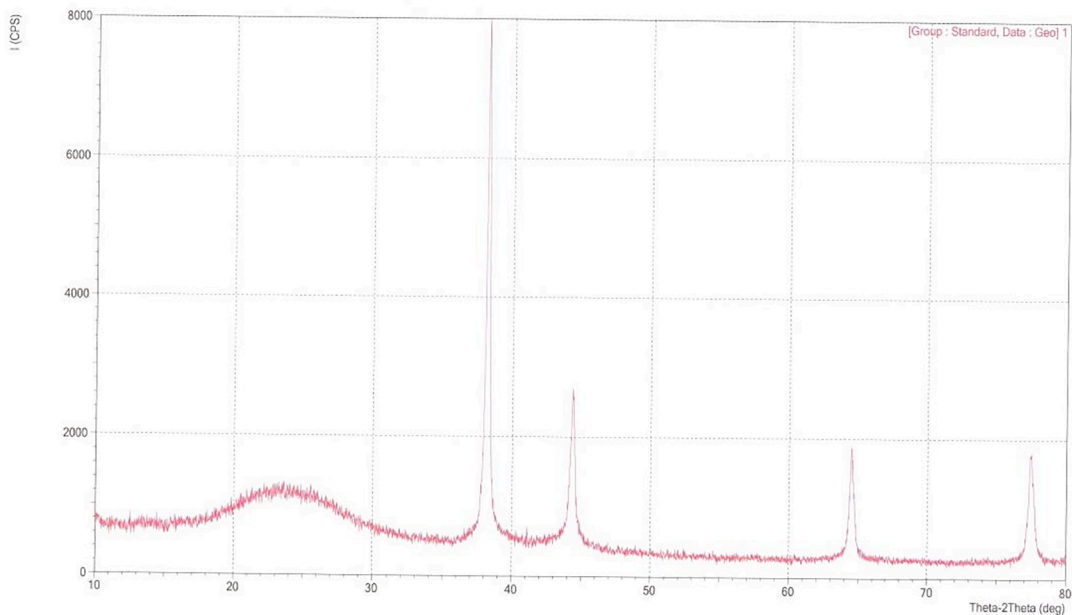
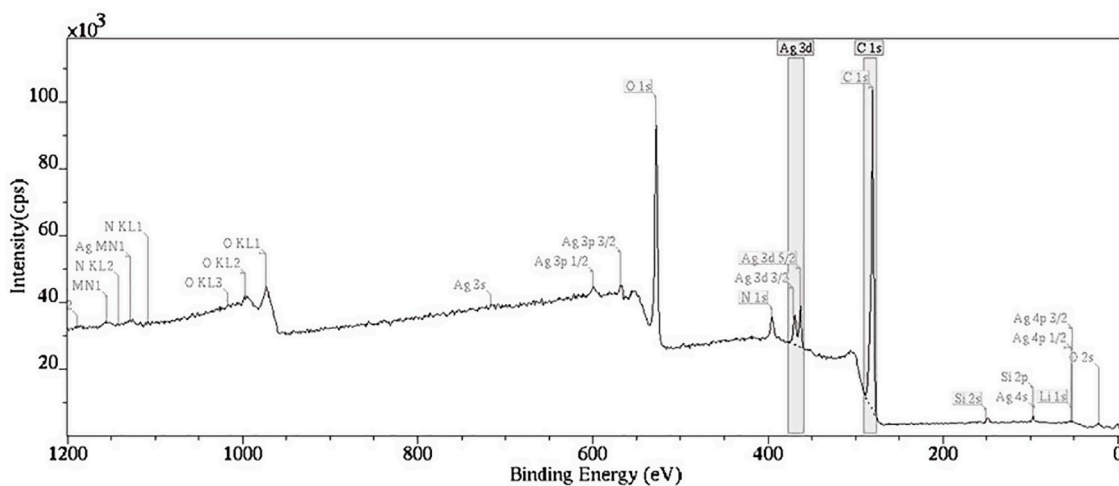


Fig. 5. XRD pattern of prepared AC after loading with Ag NPs.



State #0 : Etch Time 0.00 seconds

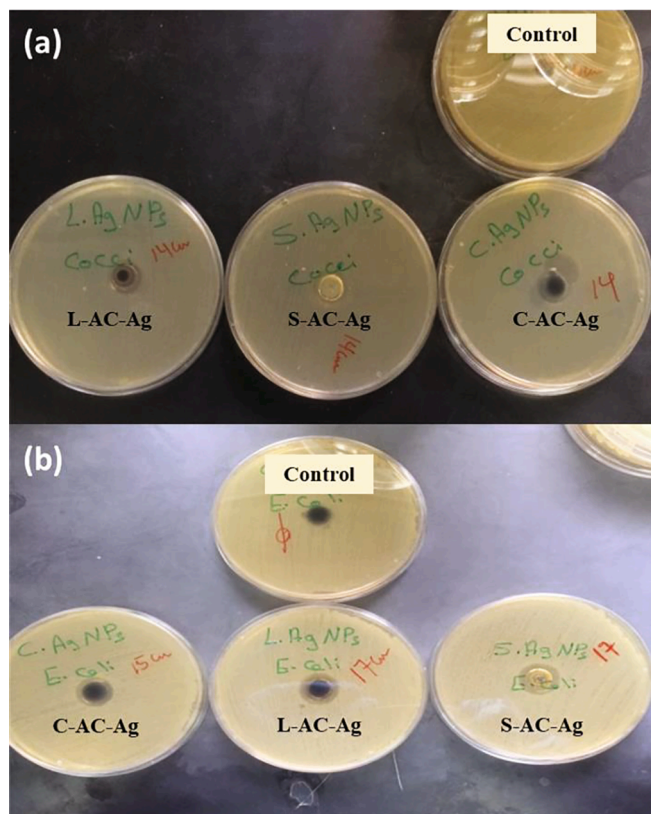
Peak	Type	Position BE (eV)	FWHM (eV)	Raw Area (cps eV)	RSF	Atomic Mass	Atomic Conc %	Mass Conc %
C 1s	Reg	281.000	3.288	381785.0	0.278	12.011	78.16	65.93
Ag 3d	Reg	364.000	3.170	74600.0	5.387	107.878	0.67	5.08
O 1s	Reg	528.000	3.860	269550.0	0.780	15.999	19.40	21.80
Si 2p	Reg	98.000	2.862	6772.5	0.328	28.086	1.10	2.17

Fig. 6. XPS survey spectrum of Ag on AC surface.

Table 1

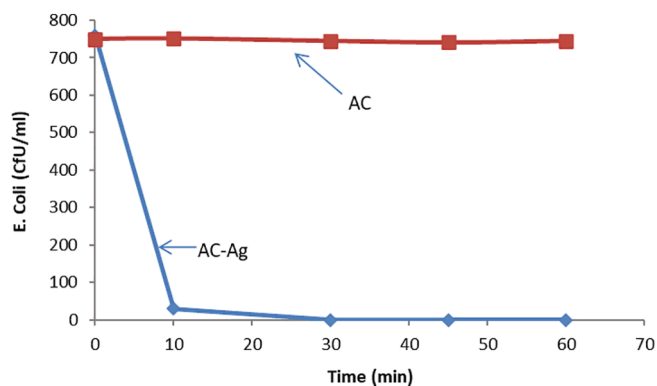
Functional groups on AC loaded and unloaded with Ag NPs.

AC type	pH _{pzc}	Basic groups (meq/g)	Carboxyl group (meq/g)	Phenol groups (meq/g)	Lacton groups (meq/g)	Total acid groups (meq/g)
AC _{unloaded}	4.2	0.09	1.62	0.32	0.9	2.84
AC _{Ag} loaded	3.9	0.07	1.57	0.25	1.15	2.97

**Fig. 7.** Inhibition zone of Staphylococci (a) and E. Coli (b) on AC loaded and unloaded (control) with Ag NPs.

with energy band gap varies between 3.1 and 3.5 eV depending on the presence of ash (Ania et al., 2012). Recent reports showed that AC could absorb UV and visible light radiation (Velo-Gala et al., 2013; Velasco et al., 2014). Therefore, exposing AC to the UV radiation should enhance and activate silver ion reduction. The reductive properties of the AC surface are confirmed by previous reports (Jeguirim et al., 2018; Aldeguer Esquerdo et al., 2021; Huang et al., 2021).

It is possible for Ag⁺ to spontaneously be reduced on the surface of AC since electrons can be donated from carbon surface which has a lower reduction potential. This could be confirmed by the presence of metallic silver on the AC surface and from X-ray analyses as shown in Fig. 5. The figure shows the major peaks for 2θ of 34.5°, 44.5°, 64.8°, and 77.6° that can be assigned to (111), (200), (220), and (311) lattice planes of FCC metallic Ag according to the JCPDF card No. 04-0783 [30]. The presence of Ag⁰ was also confirmed by the XPS measurement as shown in Fig. 6. The figure reveals the presence of the 3d5/2 and 3d3/2 states at 368.2 and 374.6 eV, respectively. The lower intensity of the 3d5/2 state indicates the presence of Ag(I). Additionally, the reduction of silver ion to metallic silver is elucidated from chemical analyses of the surface functional groups (oxidation of the OH group on the AC surface) as demonstrated in Table 1. The table shows an increase of the C=O group and a decrease of -OH group from after silver

**Fig. 8.** E. coli inactivation by AC with and without Ag NPs in batch system.**Table 2**

Removal percentage of DBPs by AC-Ag NPs.

DBPs initial concentration (mg/L)	Bromate removal (%)	Chlorate removal (%)	Chlorite removal (%)	Iodate removal (%)
10	100	97	38	62
1	97	98	31	56
0.1	99	100	60	80

impregnation.

3.2. Antibacterial effect of Ag impregnated AC

The antibacterial effect was studied by the inhibition zone and inactivation rate method (Srinivasan et al., 2013; Srinivasan et al., 2013) for two type of bacteria E-Coli (gram negative) and Enterococci Faecalis (gram positive). The results of inhibition zone method are shown in Fig. 7. Tests were conducted using commercial activated carbon (C-AC), commercial activated carbon loaded with silver NPs (C-AC-Ag), activated carbon made of palm leaves loaded with Ag NPs (L-AC-Ag) and activated carbon made of date palm pits loaded with silver NPs (S-AC-Ag). Growth of E-Coli and Cocci was inhibited around the AC-Ag samples, but not around AC alone. The inhibition zone diameter was larger for E-Coli (17 mm) than Cocci (14 mm) for all types of AC-Ag samples.

The inactivation rate method was also used to check for antibacterial effect using Ag NPs. The bacteria were completely inactivated in 30 min by Ag loaded AC, but the number of live bacteria did not change after 1 h for samples taken from AC without Ag as shown in Fig. 8.

3.3. Removal of DBPs using Ag impregnated AC

The prepared AC loaded with Ag NPs was tested for the removal of DBPs in a batch system. The results are shown in Table 2. The table shows that inorganic bromate and chlorate were completely removed after 24 h of equilibration with Ag NPs loaded AC with concentration in the range 0.1 to 10 mg/L.

4. Conclusion

Activated carbon was prepared from date palm leaves by phosphoric acid activation. Metallic silver nanoparticles were loaded by ion exchange and in-situ reduction of adsorbed silver ions under UV-radiation without using external reducing agents. Instead, phosphoric acid was used for activation. The greenness of the Ag nanoparticles on activated carbon from in-situ reduction without waste generated from the preparation processes. The disinfecting ability of the Ag – activated carbon for E-Coli and Cocci was confirmed by inhibition zone and inactivation of suspended bacteria in a batch system. The results reveal significant

enhancement of the antibacterial effect of activated carbon loaded with Ag nanoparticles which can be utilized for practical applications. The prepared AC-Ag material also has the ability to degrade bromate and chlorate to a high level. In forthcoming works, the produced AC-Ag with a suitable mesh size will be tested as a filter for drinking water disinfection and removal of DBPs. This type of filters could be used in the places where access for safe drinking water is not available. In addition, different parts of the date palm tree such as sheath fibres, shell, kernel shell, and seeds will be used for preparing AC-Ag and will be compared with AC-Ag from palm leaves as well.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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