# High Branched Precursor for Synthesis of Silver Nanoparticles and its Application to Cotton Fabric

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**H**EREIN we present a new green highly branched polymer precursor, namely starch-polyacrylic acid graft copolymer for synthesis of silver nanoparticles (AgNPs) via involvement of the copolymer in dual reduction of Ag<sup>+</sup> and stabilization of the formed AgNPs. Assessment of the formation of AgNPs was realized through submitting the silver colloidal solution of UV-vis spectral analysis; meanwhile the transmission electron microscope (TEM) was employed for measurement of the size of the formed AgNPs. Factors associated with the synthesis were investigated for the sake of optimization. For industrial applications, optimization could be achieved using AgNPs colloidal solution at a concentration of 1080ppm. The work was further extended to include treatment of cotton fabric using the so synthesis AgNPs colloidal solution as per the pad-dry-cure method in presence of a binder. Evaluation of these treated fabrics was performed through monitoring FTIR spectroscopy, wrinkle recovery angle and tensile strength in addition to bio-assay for antimicrobial activity. Thus processed fabric acquires multifunctional properties which advocate them for production of smart and intelligent textiles.

**Keywords:** Starch grafted with polyacrylic acid, Silver nanoparticales, Antimicrobial activity, Multifunctional of cotton fabric and Smart textiles.

Starch and polysaccharides found a great interest as a template for nanoparticales preparation within environment. Friendly approaches, needless to say, starch is a promising natural, biocompatible, nontoxic, and biodegradable polysaccharide, found mainly in the plants such as corn, potatoes, wheat and rice<sup>(1,2)</sup>. In addition, they are renewable and available at low cost<sup>(3)</sup>, which makes it suitable for biomedical application and favored over synthetic polymers<sup>(4)</sup>. Improvement of the functional properties of native starches is frequently achieved by modifying their physical and /or chemical structures through various chemical treatments so as to yield the claims of product characterization in many industries and application<sup>(5)</sup>.

Nanotechnology is defined as the utilization of structures with at least one dimension of nanometer size for the construction of materials. Nanotechnology not only produces small structures, but also an anticipated manufacturing technology through inexpensive control of the structure of matter. Nanoparticles commonly used in commercial product are in range of 1-100 nm.<sup>(6)</sup>. Efficiency of

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nano-silver particles as well as catalytic materials depend on their structure, shape, size distribution, and chemical-physical environment; therefore, a large spectrum of research has been focused on controlling the size and shape especially on the influencing factors of silver nanoparticle formation which are crucial in their efficiency<sup>(7,8)</sup>.

Silver nitrate is commonly used as an antimicrobial  $agent^{(9,10)}$  but it is not appropriate for the application to textile materials as it stains to black-brown when exposed to air and light due to uncontrolled reduction processes<sup>(11)</sup>.

However, a desirable level of antimicrobial activity without significant color change can be obtained with silver nanoparticles (AgNPs) deposited on the textile materials. The application of AgNPs to cotton fabrics received a great deal of attention particularly because of their high resistance to microbes.

The present work was undertaken with a view to firstly synthesize water soluble starch-Polyacrylic acid copolymer which was used in the preparation of the copolymer colloid loaded silver nanoparticls (AgNPs) in a second step. Application of such colloidal solution in presence of binder to cotton fabric via coating resulted in multi functionalized cotton fabrics as shown by enhancement of wrinkle recovery angle (WRA), tensile strength, antimicrobial activity and homogenous structure of the so processed cotton fabric. Sophisticated tools such as Uv-vis spectroscopy, TEM, SEM, along with conventional methods were used for evaluation of the multi functionalized or what is called smart textiles.

#### Experimental

#### Material

Egyptian native maize starch was supplied by Egyptian Starch and Glucose Manufacturing Company, Cairo Egypt.

Silver nitrate AgNO3 (99%) from Sigma-Alderich/Germany, Binder (Printofix Binder MTB EG liquid based on acrylate) was supplied by Clariant, Cairo Egypt. Acrylic acid (AA) and other chemicals namely :sodium perborate (spb), thiourea (TU), sodium hydroxide and sulfuric acid were of laboratory grade.

# Synthesis of starch grafted with polyacrylic acid (starch-PAA graft copolymer)

Unless otherwise indicated maize starch of known weight (100g) was put into a reaction glass vessel containing distilled water followed by gradual addition of alkali solution (50mmole/100g starch). The reaction vessel was then placed in a thermostatic water bath at  $50^{\circ}$ C. The contents, of the vessel were kept under continuous stirring for 1 hr. At the end, the pH of the reaction medium was turned to the acidic range by the addition of dilute sulfuric acid equivalent to the amount of the alkali used in the aforementioned alkali treatment. This was followed by subsequent addition of spb 10mmole/100g starch, acrylic acid 30% OWS (owing to weight of substrate) and TU 8 mmole/100g starch. A material to

liquor ratio 1:2.5 was used. The whole course of polymerization was carried out under constant stirring.

## Synthesis of silver nanoparticles

Silver nanoparticles were prepared by a simple wet chemical method. 0.3g from dry grafted starch powder was weighted and put in 95ml distilled water and the pH was adjusted at 10 using dilute sodium hydroxide. The temperature was adjusted at 70<sup>o</sup>C then 1ml silver nitrate (0.1mmole) was added dropwise while keeping the system under magnetic stirring for 1 hr.

# Application of prepared AgNPs to cotton fabric as antimicrobial agent

The cotton fabric was treated with finishing bath containing grafted starch loaded with AgNPs in presence of binder (printofix Binder MTB EG liquid). The treatment was carried out by padding the fabric in the finishing bath to wet pick up of 100%, drying at 80°C for 3 min and curing at 140°C for 3 min. The treated fabric was investigated by SEM and in addition to its antibacterial properties.

# Characterization techniques of silver nanoparticles

# Ultraviolet-visible (UV-vis) Spectral

UV-vis spectra have been proved to be quite sensitive to the formation of silver colloids because AgNPs exhibit an intense absorption peak due to the surface Plasmon excitation which describes the collective excitation of conductive electrons in a metal. AgNPs embedded in grafted starch were recorded in spectra 50 ANALYTIKA JENA SPECTROPHOTOMETER from 300 to 550. Distilled water was used as the blank.

## Transmission Electron Microscopy (TEM)

Shape and size of AgNPs were practically determined using TEM; JEOL-JEM-1200. Specimens for TEM measurements were prepared by placing a drop of colloidal solution on 400 mesh copper grid coated by an amorphous carbon film and evaporating the solvent in air at room temperature. The average diameter of the prepared AgNPs was determined from the diameter of 100 nanoparticles found in several arbitrarily chosen areas in enlarged microphotographs.

#### Scanning Electron Microscopy (SEM)

The treated fabrics were studied using a scanning electron probe microanalyzer (type JXA-840A) – Japan. The specimens were mounted on the specimen stabs and coated with thin film of gold by the sputtering method. The micrographs were taken at two magnifications, namely 1000 and 2500, using 30 kV accelerating voltage.

#### Antimicrobial activity

The antimicrobial activity of the treated fabrics was examined on *Staphylococcus aureus*, and *Escherechia coli*, by antimicrobial agar diffusion test according to AATCC Test Method 147-1988.

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# FTIR Spectroscopy

FTIR spectroscopy was measured using FTIR –FT-Raman, model: Nexus 670 (Nicollet-Madison-WI-USA). The starch (native, grafted and AgNPs) were mixed with KBr. The spectral range was 400-4000Cm<sup>-1</sup>.

#### **Results and Discussion**

### Reaction mechanisms involved in formation of AgNPs using St-g-PAA

Previous reports<sup>(12-14)</sup> have disclosed that the solution of certain polymers can be used for the synthesis and stabilization of nanoparticles. Carbohydrates of polymeric nature have been successfully used for synthesis of metal nanoparticles. Grafted polyacrylic starch macromolecules (st-g-PAA) present interesting dynamic supramolecular association facilitated by inter-and intramolecular hydrogen bonding resulting in molecular level capsules, which can act as templates for nanoparticles growth.

St-g-PAA consists of chemically modified linear polymeric polysacchairde (amylase) and highly branched one (amylopectin)(1,2), in addition to reducing aldehydic end groups. All these components with their anionic and reducing properties support the utilization of grafted starch as reducing and stabilizing agent for the synthesis of silver nanoparticles. The negatively charged soluble St-g-PAA facilitates the attraction of the positively charged silver cations to the polymeric chains followed by reduction with the existing reducing groups<sup>(15)</sup>.

Synthesis of AgNPs occurs in two steps: a) a portion of metal ions in a solution is reduced by a suitable reducing agent, b) the atoms thus produced act as nucleation centers and catalyze the reduction of the remaining metal ions present in the bulk solution. As a subsequence, the atoms coalesce leading to the formation of metal clustres since the binding energy between two metal atoms associates with excess ions. The surface ions are again reduced and in this way the aggregation process does not cease until high values of nuclearity are attained, which results in larger particles. The process is stabilized by the interaction with the polymer so preventing further coalescence and aggregation<sup>(12)</sup>.

#### Silver concentration

Figure 1(a) shows the UV-vis absorption spectroscopy for AgNPs colloidal solution prepared as described in the experimental section. As evident, the band becomes stronger and more symmetrical with a pronounced bell shape at  $\lambda$  max 415nm. The band can be assigned to the Plasmon resonance of AgNPs and verified formation of the latter.

Figure 1(b) shows the TEM of AgNPs. This figure as well as the former figure obtained figures indicate that the resultant products contain a well-stabilized AgNPs solution with a concentration of 100ppm and a diameter range of (9-16nm).

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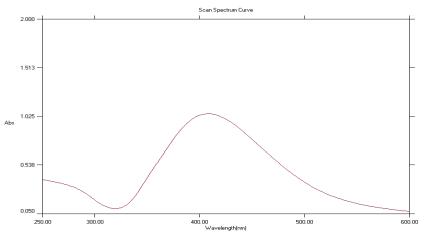


Fig. 1(a) Preparation of AgNPs; using grafted starch (a) UV-Vis absorbance of colloidal solutions using 0.3 g dry grafted starch and 1 m mole/l(100ppm) AgNO<sub>3</sub> at 70<sup>o</sup>C.

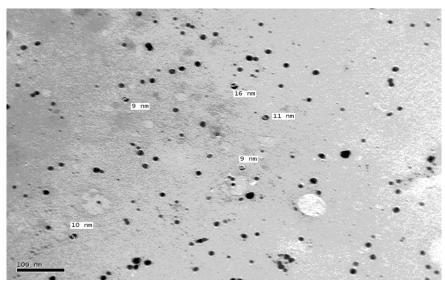


Fig. 1 (b) TEM image for AgNPs prepared at 70 °C using 1 m mole/l AgNO<sub>3</sub>.

Figure 2(a) shows the UV-vis absorption spectroscopy for AgNPs colloidal solution prepared using concentration of (1000ppm) as follows: 0.75 g of dry St-g-PAA was weighed and put in 95ml distilled water followed by pH adjustment at 10 using dilute sodium hydroxide solution and temperature adjustment at  $70^{\circ}$ C. 10 ml silver nitrate (0.1mmole) was then added dropwise while keeping the system under magnetic stirring for 1 hr. It is clear from Fig. 2(a) that the band is symmetrical but broad which could be associated with the aggregation of silver ions. This is also proved by TEM in Fig. 2(b)which shows the aggregation of this silver ions but the size still in a nano form range of (9-22nm).

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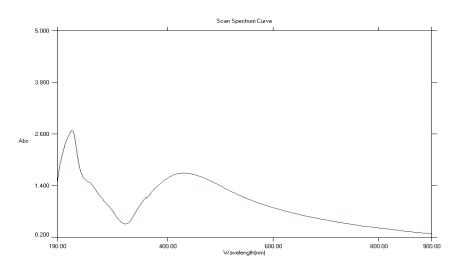


Fig. 2. Preparation of AgNPs using grafted starch ; (a) UV-Vis absorbance of colloidal solutions using 0.75g dry grafted starch and 10 m mole/l(1000ppm) AgNO<sub>3</sub> at 70  $^\circ C$ .

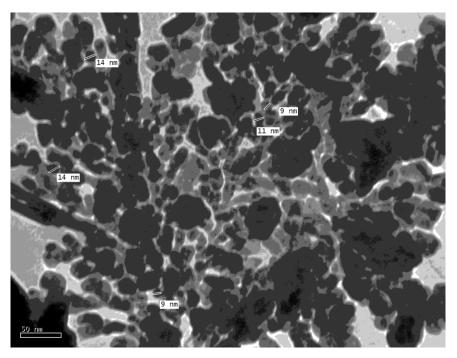


Fig. 2 (b) TEM image for AgNPs prepared using 0.75 g and 10 m mole/l(1000m mole) AgNO\_3 at 70  $^\circ C$  .

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#### FTIR spectroscopy

The role of St-g-PAA as reducing and capping agent was confirmed by FT-IR analysis of the prepared AgNPs and also for untreated and grafted starch. Figure 3(a, b, and c) shows the absorption peak at 3430cm<sup>-1</sup> corresponding to the hydrogen –bonded hydroxyl (OH) and the peak at 2923cm<sup>-1</sup> indicates the presence of C-H. The absorption peaks situated around 1738cm<sup>-1</sup>, 1626cm<sup>-1</sup> and 1460cm<sup>-1</sup> are the characteristic peaks for the C-H, C-C and C-O stretching, respectively. The absorption bands at 616,666 and 708cm<sup>-1</sup> represent the Ag in AgNPs colloidal solution<sup>(16)</sup>. Further the absorption peak appears in Fig. 3(c) at 1096 and 1046cm<sup>-1</sup> points towards the formation of a new C=O groups. This state of affairs is due to the reduction of Ag by some hydroxyl groups (in carboxylic groups) that are oxidized at the expense of Ag when Ag is reduced<sup>(17)</sup>.

# Application of silver nanoparticles (AgNPs) to cotton fabric to induce multifunctionalyzation of cotton

Cotton fibers are still by far the most important fabrics material for the production of textiles. It has excellent moisture absorption ability, which is strong in wet and dry state, and outstanding air permeability. However, cotton suffers from a number of short comings the most important of which are inability of share handing and can be easily attacked by bacteria particularly in case of moist cottons. In the current study, attempts have been made to undergo treatment of cotton fabric with AgNPs colloidal solution with a view to impart antibacterial properties to the fabric and improve its tensile strength and wrinkle recovery angle (WRA). Hence the cotton fabric was treated with a finishing bath containing AgNPs colloidal solution at concentrations 50 and 100ppm in presence of binder (prinntofix Binder MTBEG liquid 1%).The treatment was performed by padding the fabric in the finishing bath to a wet pick up of 100%, drying at 80°C for 3 min and curing at  $140^{\circ}$ C for 3 min. Given below are the results obtained along with their appropriate discussion.

## The antibacterial properties

The interaction between fibers and metallic AgNPs results from:

1.Formation of chemical bond between silver and alcoholic groups of cotton and, 2.Physical adsorption of AgNPs on the fabric surface <sup>(18, 19).</sup>

The antibacterial properties of cotton fabrics treated with AgNPs colloidal solution at different concentrations are measured according to the inhibition zone method against Gram negative bacteria (E. coli) and Gram positive bacteria (S. aureus). Table 1 shows results of the inhibition zone of cotton fabric samples treated with different concentrations of AgNPs (50 and100ppm). The results clarify that all samples have inhibition zone larger than the untreated fabric. Table 1 also shows that the inhibition zone increases by increasing the AgNPs concentration. The bacterial effect of AgNPs can be attributed to the attachment of AgNPs to the surface of cell membrane disturbing permeability and respiration functions of the cell<sup>(20)</sup>. Additionally reports suggest that ionic silver strongly interacts with the thiol group of vital enzymes and inactivates them.<sup>(21,22)</sup>. The difference between gram negative and gram positive bacteria essentially rest in the structure of their respective cell walls. The layer of peptide glycan (about 20-30nm) in gram positive bacteria is thicker than gram -negative bacteria<sup>(23)</sup>. At any event, it is assured that presence of silver nanoparticles on cotton surface is necessary for inhibition of bacterial growth. The binder used binds the AgNPs overall the cotton fabric surface as well as within the fiber pores and crevices.

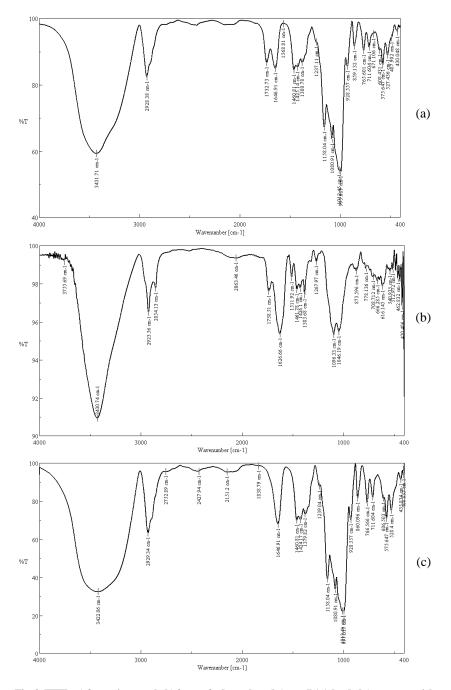


Fig. 3. FTIR; a) for native starch, b) for grafted starch and c) st-g-PAA loaded Ag nanoparticles.

 TABLE 1 Effect of AgNPs concentrations on antimicrobial activity of cotton fabric before and after washing .

	Inhibition Zone diameter (mml/1cm sample)mm				
[AgNPs] (ppm)	Escherichia coli G		Straphylococcus aureus G <sup>+</sup>		
	Before wash	After wash	Before wash	After wash	
Blank	0.0	0.0	0.0	0.0	
50	12	10	22	18	
100	13	11	23	19	

Cotton fabric treated with different concentrations of AgNPs colloidal solution to 100% wet pick –up and dried at 80°C for 3 min, then cured at 140°C for 3 min.

# SEM

Figure 4(a, b, c) shows the SEM images of the untreated fabric and those treated with AgNPs colloidal solution at concentrations of 50 and 100ppm, respectively. Figure 4(a) reflects smoothness of the untreated cotton whereas Fig. 4(b) and (c) reflect a thin film containing AgNPs. The film covers the fabric threads and coats them in a manner involving their rearrangement thereby protecting the fabric from bacterial attack. The higher the AgNPs concentration used, the more apparent is the existence of AgNPs in the SEM images.

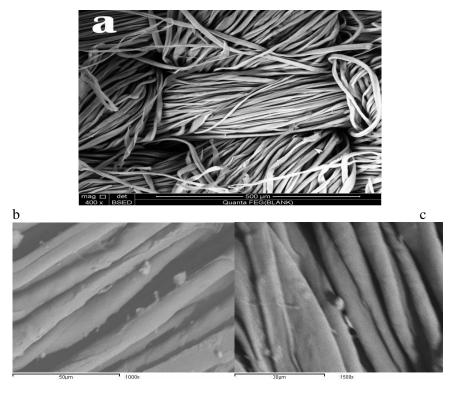


Fig. 4. SEM image of : (a) untreated cotton fabric, (b) nanosilver particles on cotton using 50 ppm, (c) nanosilver particles on cotton using 100 ppm .

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# Physical properties

It is well established that causing significant improvement in the physical properties of cotton fabric like wrinkle recovery angle (WRA) is manifestation of using proper crosslinking agent aided by appropriate catalytic system to avoid the determined effect of the whole crosslinking operation on tensile strength.

One of the most serious defects of cotton fabric. Cellulose macromolecules are composed of glucose rings which are joined together and hydroxyl groups are protruding from the macromolecular chains providing reactive cross-linking sites <sup>(24)</sup>. Cross-linking reactions occur within the accessible regions with hydroxyl groups of cellulose resulting in a better resistance to deformation and improving elastic recovery from deformation <sup>(25, 26)</sup>.

In the present work we have undertaken a novel approach for enhancement of both WRA and tensile strength. The approach is based on preparation of colloidal solutions of AgNPs and use such solutions in coating of cotton fabric. Table 2 shows that coated fabrics display much greater WRA and tensile then than the untreated fabric. Increasing the concentration of AgNPs in the colloidal solution acts in favor of these two properties. Other factors such as presence of the binder in the finishing formation along with St-g-PAA loaded silver nanoparticles and their interaction with cotton cellulose would result in cement tidal components to give ultimately cotton fabric with improved WRA and tensile strength even after washing. Indeed, the SEM images shown in Fig. 2 (b & c) present these cement tidal fibrous and nanofibrous components as highly ordered and make associated components as compared with the untreated cotton fabric.

#### Conclusion

To start with, starch grafted with polyacrylic acid (PAA) which is also well known as starch-PAA graft copolymer was synthesized under the initiation action of spb-thiourea redox system. Thus so obtained copolymer was used as reducing and stabilizing agent for AgNPs. The ultimate result was to synthesize the copolymer colloidal loaded AgNPs. In a second stage, the copolymer colloidal solution containing AgNPs was used in coating of cotton fabric and the coated fabric was evaluated as per world-class facilities as well as traditional ones. The output of the various testing and analysis pertaining to UV-vis spectral, TEM, SEM, WRA, tensile strength and, antimicrobial activity was spectacular. The use of copolymer colloidal AgNPs in presence of binder in coating cotton fabric results in multi-functionalization of the cotton fabric.

A future outlook at the present work would advocate it for further investigation including thorough evaluation as well as synthesis and characterization or other starch soluble copolymers. This is rather a start of developing a new generation of starch copolymers colloidal solutions containing metallic nanoparticles for multifunctionalization via coating of cotton fabrics production. Multifunctionalization forms, indicated, the base of smart textile.

	WRA angle		TS( Kgf)	
[AgNPs] (ppm)	Before washing	After washing	Before washing	After washing
Blank	104	103	69.5	69.5
50	220	195	124	111
100	240	210	136	120

TABLE 2. Effect of	f AgNPs concentratio	n on wrinkle recove	ry angle (WRA) and		
Tensile strength(TS) of cotton fabric before and after washing .					

Cotton fabric treated with two different concentrations of AgNPs colloidal solution to 100% pick –up and dried at  $80^{\circ}$ C for 3 min. then cured at  $140^{\circ}$ C for 3 min.

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# استخدام النشا المحور لتحضير دقائق الفضه وتطبيقه على الأقمشة. القطنية

محمد الرافعي ، منال البيسي وعلي حبيش شعبة بحوث الصناعات النسجية – المركز القومي للبحوث – القاهرة – مصر

تعتبر النشا من المواد الغذائية الرخيصة الثمن لذلك تستخدم بعد تحويرها كيميائيا باستخدام حمض الاكريليك والثيويوريا وفوق بورات الصوديوم لانتاج نشا-عديد الاكريلك سهل الذوبان في الماء الذي يستخدم لإختزال نترات الفضة وتثبيت دقائق الفضنة النانومترية المتكونة حيث يتم تتبع سير التفاعل باستخدام تحليل الموجات فوق البنفسجية المرئية وتقبيم حجم الدقائق النانومترية باستخدام جهاز الميكروسكوب الإلكتروني النافذ .

وتم معالجة الأقمشة المحتوية على السليلوز بمعلق الفضنة النانومترية وكذلك تم تقييم الأقمشة المعالجة بإستخدام الميكروسكوب الماسح الإلكتروني بالإضافة إلى خاصية مقاومة الأقمشة المعالجة للبكتريا . وكذلك تم قياس قوة الشد والاستطالة وزاوية الانفراج وثبت تحسنها بعد المعالجة . 97