

Novel Conductive Nano-composite Ink Based on Poly Aniline, Silver Nanoparticles and Nitrocellulose

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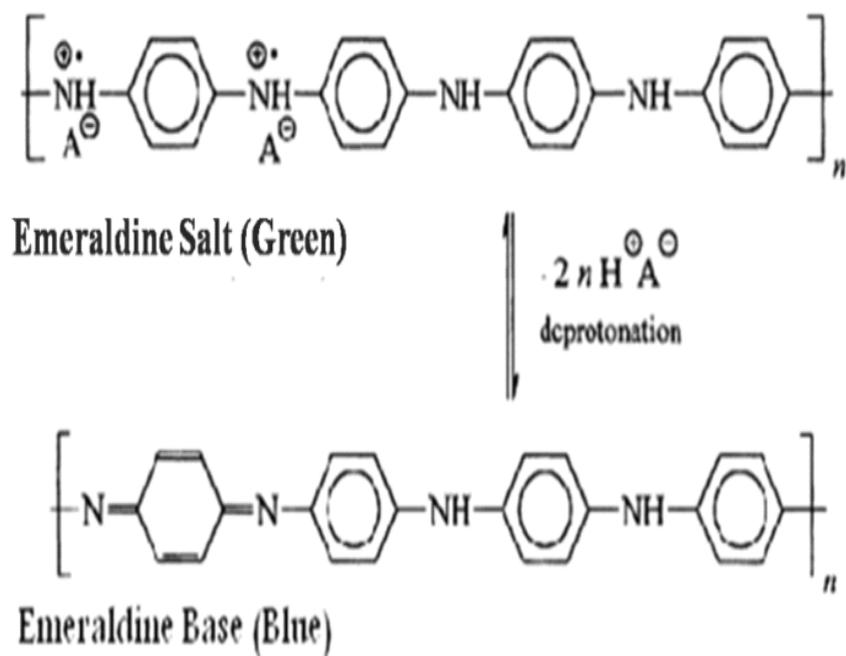
CONDUCTING polymers have attracted much attention in view of scientific interest, simple preparation method and practical applications. This article demonstrates the preparation of an electrically conductive composite based on (poly aniline, silver nanoparticles, nitrocellulose resin) by mixing the three components with each other by sonication. Poly aniline (PANI) doped with HCl was prepared by chemical oxidation pathway using potassium peroxydisulphate while silver nanoparticles (Ag-NPs) was synthesized via chemical reduction process using oleic acid and oleyl amine. The prepared materials were characterized by spectroscopic measurements (FTIR, UV-vis, TEM), in addition to thermal analysis (TGA, DSC). The electrical conductivity was measured by four-point probe method. The achieved results indicated high performance properties for the nano-composite to be applied in conducting inks.

Keywords: Conductive ink, Poly aniline hydrochloride, Silver nanoparticles, Nitrocellulose and Polymer nano composite.

In the few recent years, electrically conductive nano-inks which contain nano-sized conductive material have been attracting much attention⁽¹⁾. The electrically conductive inks are composites of the polymers and electrically conductive materials where the polymeric materials provide the mechanical and processing properties while the electrically conductive materials provide the electrical properties⁽²⁾. Polyanilines have accepted a great importance with respect to the scientists due to its simple and easy synthetic methods, raw materials availability, lower cost, light weight, good environmental stability, and corrosion resistance; in addition to its various applications⁽³⁾. Because of the different properties of PANI so it has a wide applications such as, a conducting

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polymer, efficient corrosion inhibitors for steel protection⁽⁴⁾, lightweight rechargeable battery^(5,6), and electromagnetic shielding devices^(7,8). PANI is polymerized by both chemical and electrochemical polymerization⁽⁹⁾. The synthetic methods of conducting polymers, especially polyaniline include chemical and electrochemical pathways and it has a very simple acid/base doping/dedoping chemistry⁽¹⁰⁻¹³⁾, as shown in Scheme (1). One of the common synthesis methods of polyaniline is, the chemical oxidation of aniline using potassium peroxydisulphate or ammonium peroxydisulphate as initiators⁽¹⁴⁾ and hydrochloric acid as dopant⁽¹⁵⁾. PANI has its importance due to its good combination of low cost, stability and conductivity⁽¹⁶⁾. However, PANI is poor processing, rigid and non-film forming polymer⁽¹⁷⁾. Many articles were published to overcome with these problems and one of these ways is applied by dispersing PANI (or any other non film forming material having electrical conductivity) in a polymeric material having good mechanical and processing properties⁽²⁾. As reported by Veluru, J. B. *et al.* where PANI was dispersed in Poly Methyl Methacrylate (PMMA)⁽¹⁸⁾. Some of the most popular polymeric materials that used as binders are poly acrylates eg. PMMA, epoxy resin^(19,20), polyurethane⁽²¹⁾, and polystyrene⁽²²⁾, where these materials have good mechanical properties and good processability.



Scheme 1. The conductive form (Emeraldine salt) and nonconductive form (Emeraldine base) of PANI⁽²³⁾

Silver nanoparticles are one of the most popular nano-materials that used to disperse in organic polymers and considered the most attracted to researchers, but there are other different nano-materials such as: grapheme⁽²⁴⁾, copper⁽²⁵⁾, carbon nano-tubes⁽²⁶⁾, oxides (e.g. TiO_2 , Fe_3O_4 , SiO_2)^(27,28) and others have been used.

Nitrocellulose is one of the oldest and most widely used film formers adaptable to a number of wide applications such as in protective and decorative coatings for wood and metal, flexible coatings for paper, foil and plastic films, printing inks, and adhesives⁽²⁹⁾. The film-forming and keying properties of nitrocellulose are outstandingly good⁽³⁰⁾. Nitrocellulose is characterized by its thermoplastic behavior, fast solvent evaporation, good compatibility with a wide range of materials, excellent processability and good mechanical properties^(29,31). Due to a unique combination of properties, celluloses are a good choice to disperse the electrically conductive materials (PANI and Ag-NPs) in it^(32,33).

The electrically conductive metallic inks have various electronic applications including flat panel displays (FPD), semiconductor packaging, photovoltaic cells, and micro-electromechanical systems (MEMS), plasma displays (PDP), radio frequency identification (RFID) and photovoltaic solar cells⁽³⁴⁾.

Generally, corporation of conducting polymer and metallic particles with polymeric materials lead to make a desirable product with integrated properties to be suitable for a wide variety applications⁽³⁵⁻³⁶⁾.

Due to the large deficiency in literature concerning the developments of conductive ink nano-composites based in cellulosic nitrate, this work was demonstrated to prepare conductive ink nano-composite based on poly aniline hydrochloride, silver nano-particles and nitrocellulose.

Materials and Methods

Materials

Aniline (99.5%, Sigma-Aldrich), potassium peroxydisulphate (98%, Labmerk Chemicals Pvt. Ltd.), hydrochloric acid (34-36%, El-Nasr for intermediate chemicals Co.), ethanol (96% absolute, ALAMIA Co.), silver nitrate ($\geq 99\%$, Sigma-Aldrich), oleyl amine and oleic acid (technical grade 70%, Sigma-Aldrich), methanol for HPLC (99.9%, Sigma-Aldrich), n-hexane (99%, Sigma-Aldrich), toluene (99.5%, Scharlau), nitrocellulose (NC) lower nitrogen content was commercial material.

Methods

In this method, PANI was synthesized firstly, then Ag-NPs was synthesized individually, then both of PANI and Ag-NPs was dispersed in suitable solvent also individually then both solutions of PANI and Ag-NPs were dispersed in nitrocellulose solution.

Synthesis of PANI conducting polymer

After twice distillation, (2 ml, 0.022 mole) of aniline was added to 10 ml absolute ethanol and placed in ice bath (0-5 °C) with continuous stirring. 5.5 ml of 0.1 M HCl solution was added then also solution of (6 g, 0.022 mole) potassium peroxydisulphate in 100 ml distilled water was added dropwise. The color of reaction medium was changed to reddish brown after addition of first 5ml of potassium peroxydisulphate solution then to dark green color. After 1 hr of continuous stirring, the reaction medium was remained to stand for 24 hr. Precipitate was separated by filtration, then washed with 200 ml warm distilled water and 100 ml ethanol and left to dry in an oven at 80 °c for 4 hr. Further doping of PANI was done in 100 ml of 2M HCl for 2 hr at room temperature and then the product was filtered and dried.

Synthesis of Ag-NPs

Silver nitrate AgNO₃ was used as a source of Ag metal particles, while a mixture of oleyl amine (OAm) and oleic acid (OAc) was used as reducing and capping agents to control both the morphology and the size of the Ag metal nanoparticles. The synthesis of Ag-NPs was as the following:

AgNO₃ (0.01 g) was dissolved in a mixture of 5 ml oleyl amine and 3 ml of oleic acid whereas the dissolution was occurring by sonication. The chemical reaction was accelerated by leaving the reaction mixture in microwave device for 1 min. The color of reaction medium changed from yellow to brown after getting out from microwave device.

10 ml of methanol was added to the mixture, then the nanoparticles were precipitated out and the flocculate was separated from the supernatant by centrifugation. Dispersion of the flocculate in 2 ml of toluene and 5 ml of methanol followed by further centrifugation. This step was repeated to obtain pure nano-particles. Finally, the precipitated nanoparticles were dispersed in 5 ml hexane, then dried at 40°C in the oven and collected as powder.

Preparation of conductive ink nano-composite (PANI, Ag-NPs, NC)

Firstly, the conductive ink nano-composite was prepared as the following: 0.1 g Ag-NPs was dispersed in chloroform, 0.04 g PANI was dispersed in chloroform, 0.2 g NC dissolved in ethanol. The dispersion occurred by sonication for 2 hr. After dispersion occurred, the three components were mixed with each other, then the mixture was sonicated for 10 min after that, the composite has been ready to drawdown by the spin coater to form film on glass slit to be ready for measuring the electrical conductivity.

Characterization

PANI was characterized with UV-vis spectroscopy, FT-IR, Thermal gravimetric analysis (TGA), Differential Scanning Calorimetry (DSC) while Ag-NPs was characterized by UV-vis and TEM whereas PANI and (PANI, Ag-NPs, NC) composite was measured by four-point probe method to assay the electrical conductivity.

The conductivity of the sample was measured by the use of four-probe technique on pellets compressed for PANI at 600 MPa, 13 mm in diameter and 1–1.5 mm thick and by using spin coater for (PANI, Ag-NPs, NC) composite, using a Keithley resistivity range 0 – 200 mega ohm and at 200 volts at room temperature. The sample of a composite for conductivity measurement was prepared by drawdown the composite solution on glass slit by spin coating technique then evaporation of solvents was occurred.

FT-IR spectra were taken with KBr pellets by using JASCO 4100-A FT-IR spectrometer, UV-vis spectra were obtained by the use of Shimadzu UV-1600 series spectrophotometer. TGA was made by using Shimadzu DTG-60H under Nitrogen Atmosphere with gas flow rate 20 ml/min at heating rate 10 °C/min in the temperature range 0 – 700 °C. Differential Scanning Calorimetry (DSC) was carried out by using Shimadzu DSC-60 under Nitrogen Atmosphere with gas flow rate 20 ml/min at heating rate 10 °C/min in the temperature range 10 – 400 °C.

The shape and size of the Ag-NPs were evidenced by JEM-2100 Transmission electron microscopy (TEM) operated at 200 kV. Sample for TEM was prepared by placing a droplet of a colloid suspension in toluene for organic media on a carbon-coated, 300-mesh copper grid (Ted Pella) and allowed to evaporate in air at room temperature.

Results and Discussion

PANI analysis

FT-IR spectroscopic analysis

Figure 1 illustrates the FTIR spectrum of PANI doped with HCl acid.

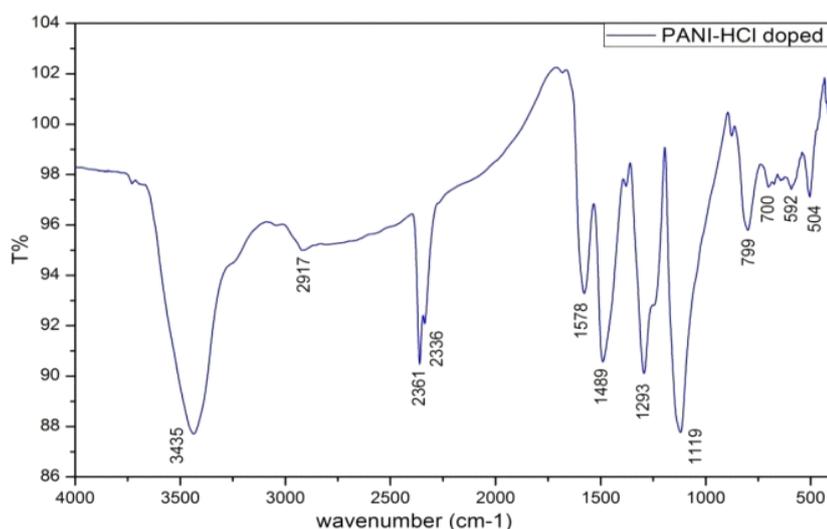


Fig. 1. FT-IR spectra of PANI doped with HCl acid.

It is clearly seen from the figure that there are characteristic absorption bands as shown in Table 1 where, N-H stretching band for secondary amine at 3435 cm^{-1} , the N-H wagging band for secondary amines appears at 700 cm^{-1} and the band at 1293 cm^{-1} represent C-N stretching bands secondary aromatic amine^(14, 37). The main bands of protonated PANI are bands at 1578 cm^{-1} and 1489 cm^{-1} can represent the stretching vibration of the quinonoid and benzenoid rings "the band at 1578 cm^{-1} arises from C = C of quinonoid rings (Q) while the band at 1489 cm^{-1} is due to vibration of C = C associated with the benzenoid ring (B)"⁽³⁸⁾. The proofing of protonation "doping" to PANI is through band appearing at 2361 cm^{-1} that present stretching vibration of (NH⁺) tertiary amine salt⁽³⁹⁾. The band indicating the polymer formation is the band located at 799 cm^{-1} that is characteristic of para distributed aromatic rings^(38, 40, 41).

The strong band at 1119 cm^{-1} and broad strong absorption tail at $2300\text{-}2700\text{ cm}^{-1}$ are appearing due to stretching vibration of B-NH⁺=Q where those two bands are considered the most important FTIR bands to evidence that high electrical conductivity and high degree of electron delocalization in polyanilines were found⁽⁴²⁾.

TABLE 1. Assignments of main FTIR bands of PANI-HCl.

Wavenumbers (cm ⁻¹)	Assignments
3435	v(N-H) stretching secondary amine
2917	v(C-H) of Aromatic ring
2361 , 2336	v(NH ⁺) tertiary amine salt
1578	v(C = C) of quinonoid rings
1489	v(C = C) of benzenoid ring
1293	v(C-N) in secondary amine
1119	v(B-NH ⁺ =Q)
1119 , 799	v of para substituent on Aromatic ring
700	N-H wagging for secondary amine

UV-vis spectroscopic analysis

Figure 2 shows the UV-vis spectrum of PANI. It can be illustrated from the figure that there are two characteristic absorption peaks; the evidence of formation of PANI conductive polymer is the max absorption peak at 206 nm that represents $\pi\rightarrow\pi^*$ transition of a conjugated ring system and absorption peak at 367 nm may be due to $n\rightarrow\pi^*$ transition of a lone pair of electron on imine. These results are in accordance with the data reported in previous study⁽⁴³⁻⁴⁹⁾.

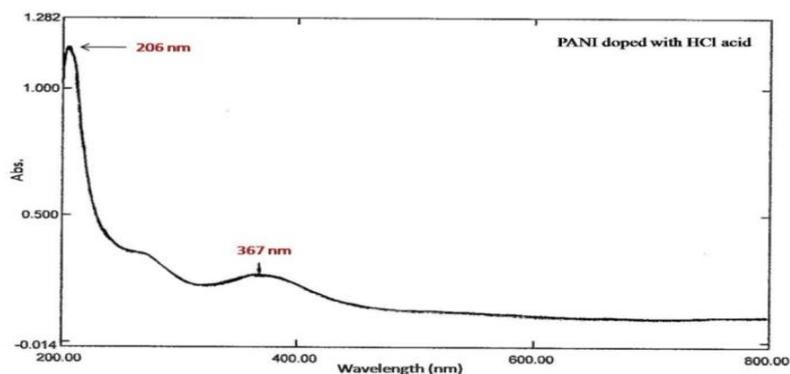


Fig. 2. UV-vis spectrum of PANI-HCl.

Thermal analysis

Thermal behavior of PANI can be identified by Thermal Gravimetric Analysis (TGA) and also by Differential Scanning Calorimetry (DSC); TGA is studying the thermal stability of PANI by measuring the weight loss of the polymer with changing the temperature and DSC is determining the glass transition temperatures (T_g)⁽³⁸⁾. The analysis occurred with measuring conditions that have been mentioned previously.

Figure 3 shows the weight loss of PANI from temperature 23 °C to 91 °C; the weight loss of PANI sample is 7 %, this may be due to absorbed moisture, from 91 °C to 292 °C the sample decomposes gradually whereas from 292 °C to 610 °C the weight of sample decreases dramatically approximately 80 % that due to degradation of polymer. So, the PANI has thermal stability up to 300 °C.

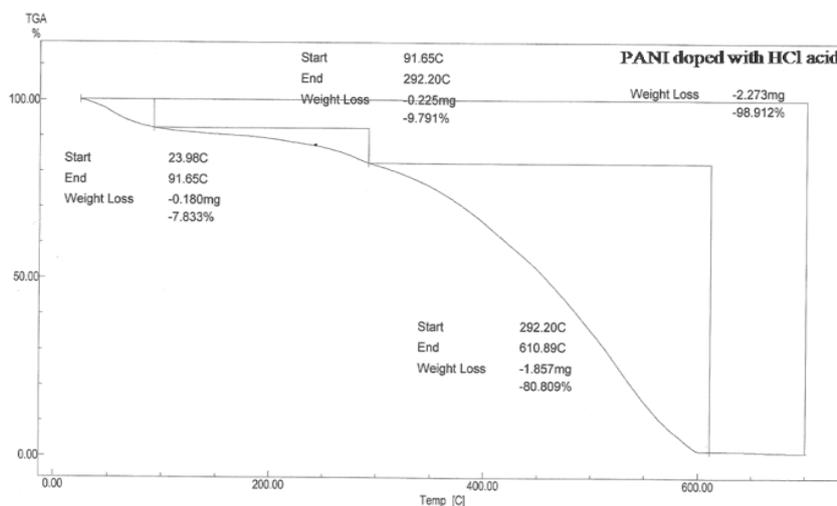


Fig. 3. TGA curve of PANI-HCl.

Figure 4 shows the DSC curve of PANI and there are two transitions on this curve; the first transition at 81 °C represents the glass transition temperature (T_g) of polymer and the second transition at 262 °C is due to the start of polymer degradation.

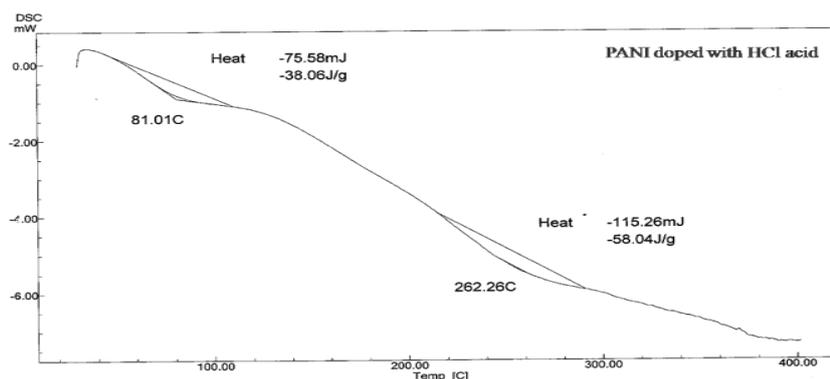


Fig. 4. DSC curve of PANI-HCl .

- *Ag-NPs Analysis*

- TEM and UV-vis.*

The max. Plasmon band at 416 nm is observed due to the formation of spherical Ag-NPs, as shown in Fig. 5. This means that (OAc-OAm) could act as both a reducing agent and a capping material⁽⁵⁰⁾. The onset of the color changes from yellow to brown corresponding to the formation of Ag-NPs. The image of TEM is an evidence for the formation of Ag-NPs as shows in Fig. 6 whereas the spherical shape shows at scale bar 20 nm, 50 nm and 100 nm. Histogram (Fig. 7) showed the particle size distribution of Ag particles and gave a particle size average approximately equal 18 nm.

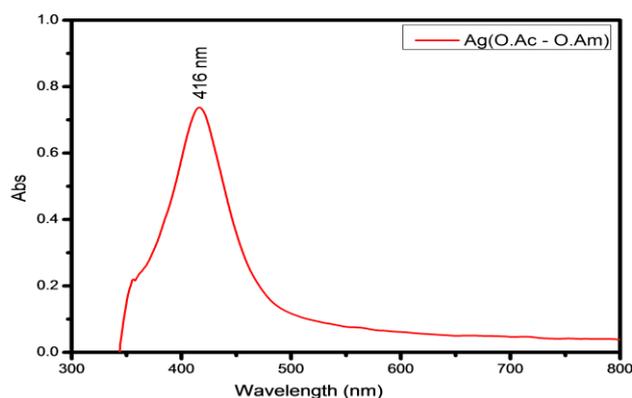


Fig. 5. UV-vis spectrum of Ag-NPs .

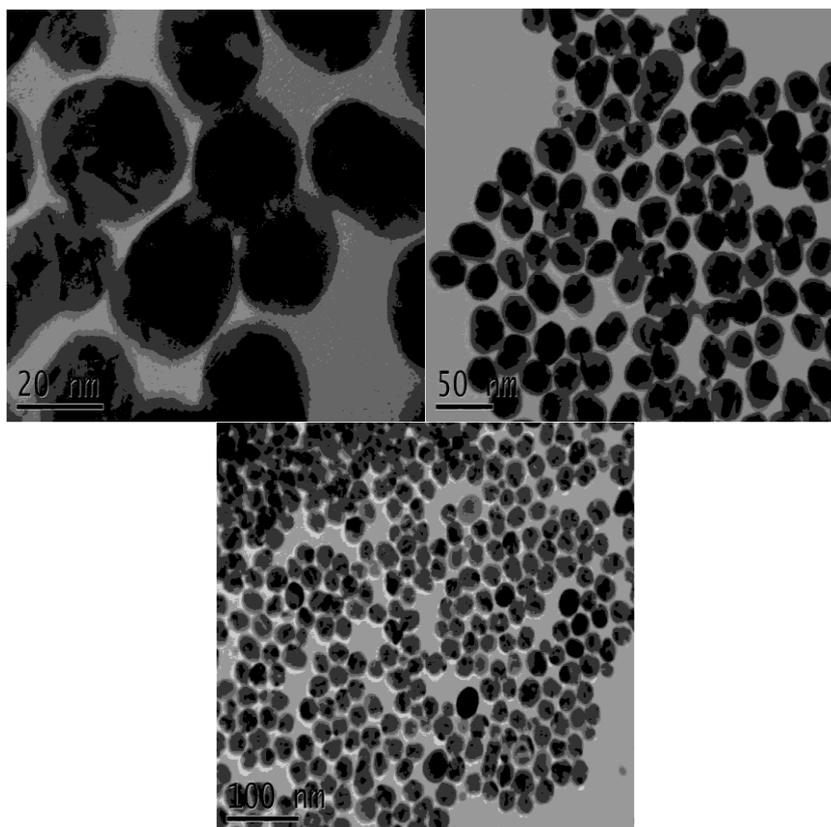


Fig. 6. TEM image of Ag-NPs (a) scale bar 20 nm, (b) 50 nm and (c) 100 nm.

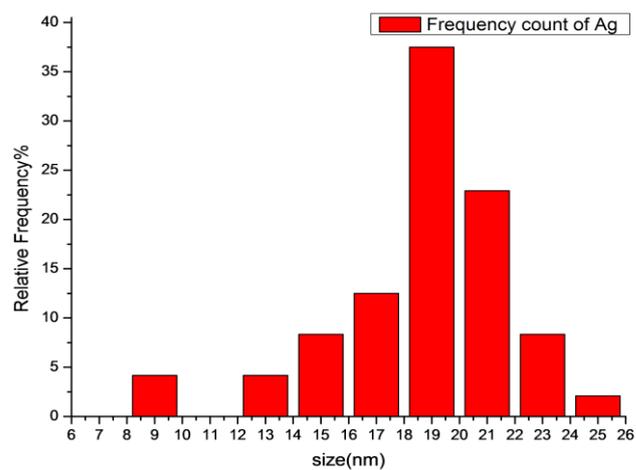


Fig. 7. Histogram shows particle size distribution of Ag-NPs .

Electrical conductivity analysis

The electrical conductivity assay of PANI pellet was 4.8×10^{-2} S/cm whereas the electrical conductivity of the film of (PANI, Ag-NPs, NC) composite was 0.6 S/cm. This result is to be considered good for this first composite made from these three components.

Conclusion

The prepared novel conductive ink nano-composite of (Poly aniline, Silver nanoparticles, Nitrocellulose) with percentage ratio (27:66:7) respectively and with spherical Ag-NPs with average particle size 18 nm, showed electrical conductivity of 0.6 S/cm. This result produces a new composite with integrated properties that can be useful for conductive ink applications and other advanced technologies.

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محمود أحمد عبد الغفار¹ ، خالد أحمد شافعي² ، أمينة فؤاد زكري²، منى بكر محمد² و خالد عبدالرازق جمعة²

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جذبت البوليمرات الموصلة للكهرباء انتباه الكثير من المهتمين علميا لبساطة طرق تحضيرها ولتطبيقاتها العملية. وتتناول هذه الدراسة تحضير متراكب موصل للكهرباء مبنيا على (البولي أنيلين ، حبيبات الفضة النانومترية، راتنج النيتروسيليلوز) من خلال خلط الثلاث مكونات مع بعضهم البعض بتقنية الموجات فوق الصوتية. البولي أنيلين المطعم بالهيدروكلوريك تم تحضيره بطريقة الأكسدة الكيميائية بينما تم تحضير حبيبات الفضة النانومترية بطريقة الإختزال الكيميائي. تم توصيف البولي أنيلين المطعم بالهيدروكلوريك بواسطة مطياف الأشعة تحت الحمراء، مطياف الأشعة المرئية وال فوق بنفسجية، التحليل الحراري الوزني وكذلك بالتحليل العياري التفاضلي؛ بينما تم التحقق من تكوين حبيبات الفضة النانومترية باستخدام مطياف الأشعة المرئية وال فوق بنفسجية وكذلك بالمجهر الإلكتروني النافذ. التوصيلية الكهربائية تم قياسها لكل من البولي أنيلين ومتراكب (البولي أنيلين، حبيبات الفضة النانومترية، راتنج النيتروسيليلوز) بطريقة النقاط الأربع. النتائج التي تحققت تكشف الخصائص عالية الأداء للمتراب النانومتري القابلة للتطبيق في الأحبار الموصلة للكهرباء.