

Original Research Article

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## Synthesis and Characterization of Polyaniline Based materials: Their biological relevance- An Overview

Sunandana Mandal\*, Swadhin Kumar Saha and Pranesh Chowdhury

Department of Chemistry, Siksha-Bhavana, Visva-Bharati, Santiniketan-731235, India

\*Corresponding author

### ABSTRACT

The present paper reveals the synthesis of polyaniline, polyaniline nanoparticles and polyaniline composite. Improvement of polyaniline properties can be achieved either by forming composites and nanocomposites of aniline, or blends with commercially available polymers or inorganic materials which offer better mechanical and optical properties, stability and process ability. Thus nanostructured (nanoparticles/-rods/-wires) conducting polyaniline offer great research interests. The synthesis of Polyaniline nanoparticles have been studied by sonochemical techniques. The synthesized nanoparticles were characterized by TEM images. The PANI/PVA/Ag nanocomposites were synthesized by chemical oxidation polymerization of aniline monomer in the presence of PVA and Ag nanoparticle colloidal solution. The antibacterial activity of the obtained nanocomposite was evaluated against Gram positive bacteria *Staphylococcus aureus* (*S. aureus*) and gram negative bacteria *Escherichia coli* (*E. Coli*) using paper disc diffusion method. The antibacterial study showed that the PANI/PVA composite did not have a very good antibacterial activity but PANI/PVA/Ag composites were found to be effective against two bacteria.

#### Keywords

Polyaniline based materials,  
Synthesis,  
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biological relevance

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### Introduction

With the discovery in 1960 of intrinsically conducting polymers (ICPs), an attractive subject of research was initiated because of the interesting properties and numerous application possibilities of ICPs. Conductive polymers have various applications such as rechargeable batteries (1), electromagnetic interferences (EMI) shielding (2), antistatic coatings<sup>3</sup>, gas sensors<sup>4</sup>, optical devices<sup>5</sup>, removal of heavy metal from water and waste water (6-7), etc. However, many of the potential uses for ICPs have yet to be explored because of a number of obstacles that need to be overcome. Among the available ICPs, polyaniline (PANI or PAn) is

found to be the most promising because of its ease of synthesis, environmental stability, low cost monomer, tunable properties, better stability compared to other ICPs, good redox reversibility and stability in aqueous solutions and air for its applications 8-9 in electrochromic displays (10), electrocatalysis<sup>11</sup>, rechargeable batteries (12-14), and sensors (15). The thermal stability of PANI is superior to other ICPs. The processability is also fairly good. All these factors contribute to PANI being superior to other ICPs. It also has a wide range of electrical properties which can be easily controlled by changing its oxidation and protonation sites (16-17).

The main problem associated with the effective utilization of all ICPs including PANI is inherent in their lower level of conductivity compared to metal, and their infusibility and poor solubility in all available solvents 18-19. There is ample scope for modifying the conductivity and processability of PANI through the selection of a suitable dopant and suitable level of doping and also by controlling its structure during synthesis (20-21). Another avenue for the successful utilization of PANI is through blending it with a commercially available polymer that has good processability and mechanical properties.

ICPs are inherently conducting in nature due to the presence of a conjugated electron system in their structure. A high level of conductivity (near metallic) can be achieved in ICPs through oxidation–reduction as well as doping with a suitable dopant 22=23. The first ICP to be discovered was polyacetylene, synthesized by Shirakawa Louis *et al.*, 24. Following the study on polyacetylene, other polymers such as polypyrrole (PPY), polythiophene, PANI, poly(p-phenylenevinylene), and poly(p-phenylene), as well as their derivatives, have been synthesized and reported as a new group of polymers known as ICPs. The conductivity of doped polyacetylene is comparable with that of metallic copper but its stability and processability are very poor compared to normal polymer (Fig. 2) even with respect to those shown in Fig. 1. The conductivity of polypyrrole, polythiophene and PANI is comparatively less but these polymers have better stability and processability compared to those of polyacetylene and polyphenylene.

The main polyaniline structure is in Fig.3, in which  $n+m = 1$ ,  $x =$  half degree of polymerization.

Polyaniline can be found in one of the three

following idealized oxidation states (25), which are polymerized from the inexpensive aniline monomer (Fig.4):

Leucoemeraldine – white/clear & colorless ( $C_6H_4NH$ )<sub>n</sub> with  $n = 1$ ,  $m = 0$  is the fully reduced state.

Pernigraniline– blue/violet ( $C_6H_4N$ )<sub>n</sub> is the fully oxidized state ( $n = 0$ ,  $m = 1$ ) with imine links instead of amine links. Studies have shown that most forms of polyaniline are one of the three states or physical mixtures of these components.

Emeraldine– green for the emeraldine salt, blue for the emeraldine base ( $[C_6H_4NH]_2[C_6H_4N]_2$ )<sub>n</sub> with ( $n = m = 0.5$ ) often referred to as emeraldine base (EB), is neutral; if doped (protonated) it is called emeraldine salt (ES), with the imine nitrogens protonated by an acid.

Emeraldine base is regarded as the most useful form of polyaniline due to its high stability at room temperature and the fact that, upon doping with acid, the resulting emeraldine salt form of polyaniline is highly electrically conducting<sup>26</sup>. Leucoemeraldine and pernigraniline are poor conductors, even when doped with an acid.

The colour change associated with polyaniline in different oxidation states (Fig.5) can be used in sensors and electrochromic devices<sup>27</sup>. Although colour is useful, the best method for making a polyaniline sensor is arguably to take advantage of the dramatic changes in electrical conductivity between the different oxidation states or doping levels<sup>28</sup>. Treatment of emeraldine with acids increases the electrical conductivity by ten orders of magnitude. Undoped polyaniline has a conductivity of  $6.28 \times 10^{-9}$  S/m, while conductivities of  $4.60 \times 10^{-5}$  S/m can be achieved by doping to 4% HBr<sup>29</sup>. The same

material can be prepared by oxidation of leucoemeraldine.

Polyaniline is more noble than copper and slightly less noble than silver which is the basis for its broad use in printed circuit board manufacturing (as a final finish) and in corrosion protection (30).

Improvement of polyaniline properties can be achieved either by forming composites and nanocomposites of aniline, or blends with commercially available polymers or inorganic materials which offer better mechanical and optical properties, stability and processability (31-34). Thus nanostructured (nanoparticles/-rods/-wires) conducting polyaniline with unusual physical and chemical properties have attracted great research interests. Much research has been conducted on the nanostructure of polyaniline (PANI) because it exhibits enhanced performance in applications where a high surface contact area

is needed between the nanostructures and its environment. Recently various strategies including sonochemical synthesis, template synthesis, interfacial polymerization, self-assembly and stepwise electrochemical deposition have been developed for the preparation of polyaniline nanostructures (35-39). Although various methods of preparing PANI nanostructures have been reported, new simple and economic methods are still being explored.

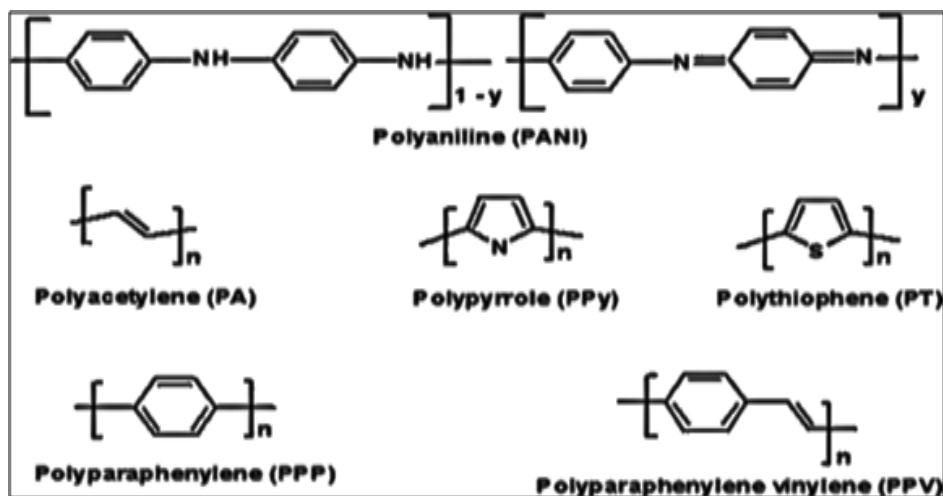
### The main objectives of the present study reflects

Synthesis and characterization of Polyaniline based materials

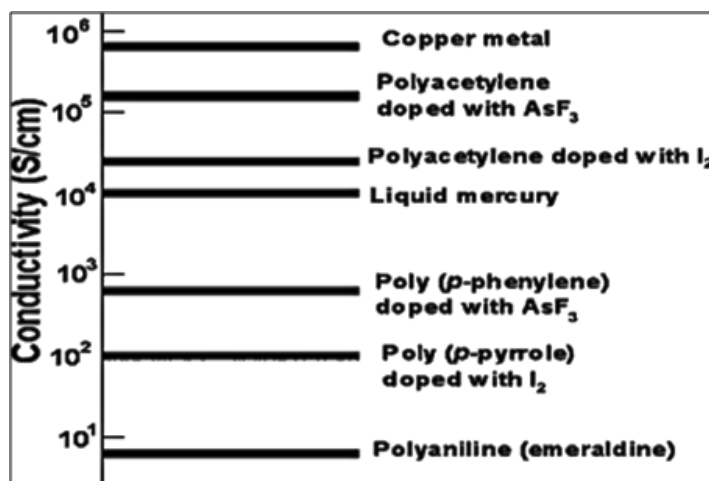
Study of their chemical and electrochemical properties

Searching for biological relevance

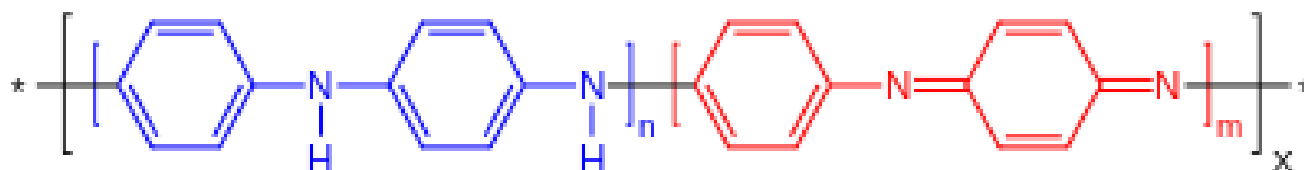
**Fig.1** The structure of a number of intrinsically conducting polymers (ICP)



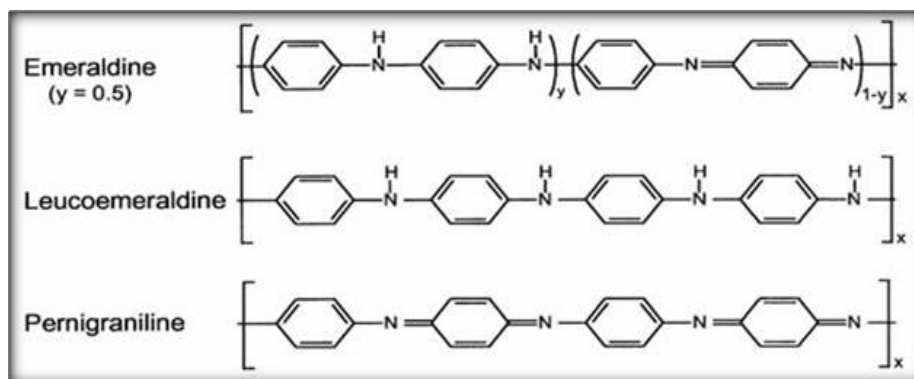
**Fig.2** The conductivity of a number of ICPs relative to copper and liquid mercury



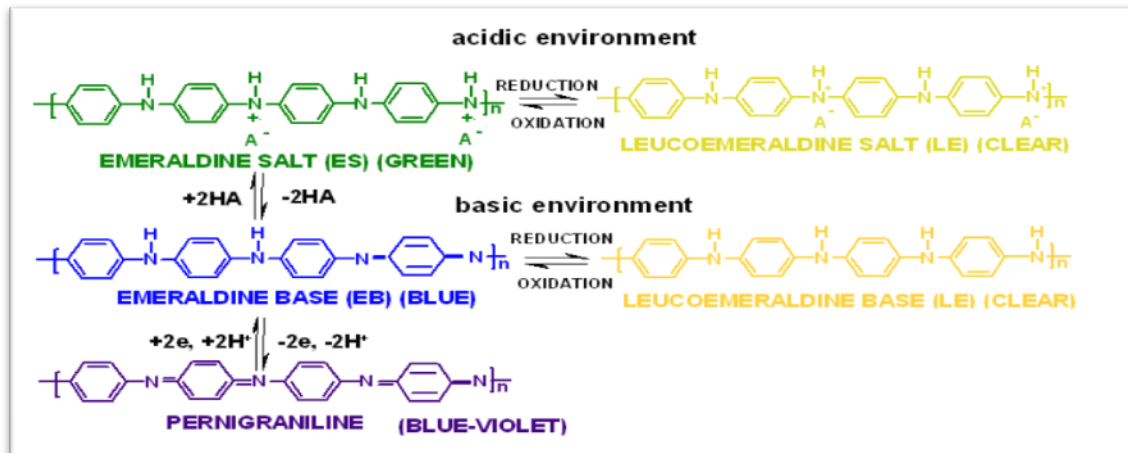
**Fig.3** Main polyaniline structure



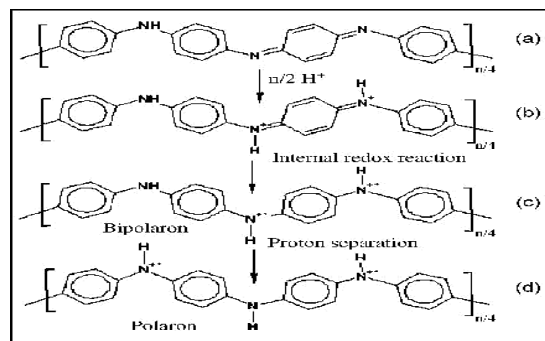
**Fig.4** Three forms of PANI



**Fig.5** Colour change associated with polyaniline in different oxidation states



**Fig.6** Sketch of Emeraldine Base (EB) before Protonation (a); after 50% protonation (b); Formation of Bipolaron (c); and Polaron (d)



**Fig.7.** Polaron and Bipolarons in PANI structures

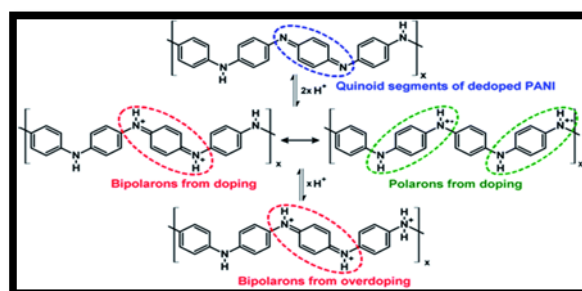


Fig.8 Shimadzu UV-VIS Spectrophotometer



Fig.9 Sonicator (Branson 1510)



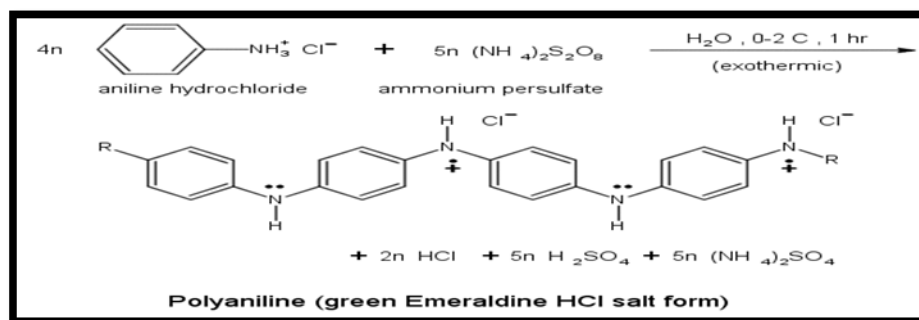
Fig.10 High resolution Transmission Electron Microscope (HRTEM) (JEM-2100, 200kv, jeol)



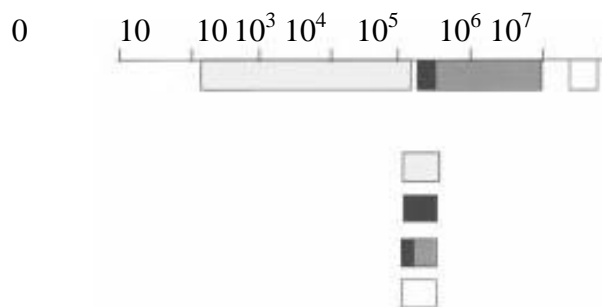
Fig.11 Centrifuge Machine



Aniline hydrochloride and Ammonium Persulfate reacts to form Emeraldine

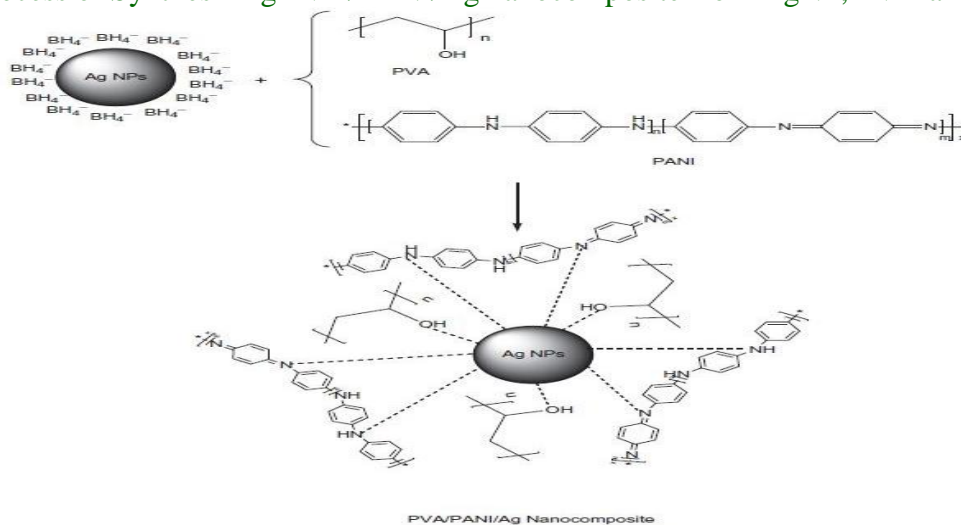


**Fig.12** Sound frequency ranges

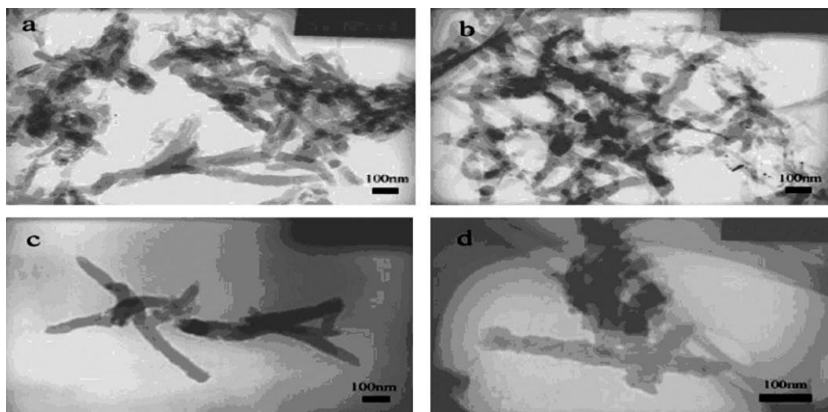


Human hearing 16Hz-18kHz  
 Conventional power ultrasound 20kHz-40kHz  
 Range for sonochemistry 20kHz-2MHz  
 Diagnostic ultrasound 5MHz-10MHz

**Fig.13** Process of Synthesizing PVA/PANI/Ag nanocomposite from AgNP, PVA and PANI



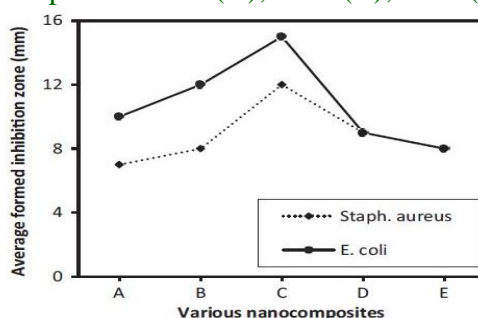
**Fig.14** TEM images of the sonochemical synthesized PANI nanofibers with APS/Aniline molar ratios of (a) 0.5, (b) 1.0, (c) 1.5 and (d) 2.0. (Reproduced with permission from Jing *et al.*, [44])



**Fig.15.** Antibacterial activity of various nanocomposites against two pathogenic strains; *E. coli* and *Staph. aureus* shown by the paper disk diffusion method; 5% (A); 10% (B); 15% (C); 20% (D); 25% (E)



**Fig.16** Scatter plot of inhibition zone versus various PVA/PANI/Ag nanocomposites at different concentrations of Ag nanoparticles: 5% (A); 10% (B); 15% (C); 20% (D); 25% (E)



**Table.1** Average inhibition zones obtained from various nanocomposites at different concentrations of silver nanoparticles; 5% (A); 10% (B); 15% (C); 20% (D); 25% (E); 0% (F) against two pathogenic bacteria

Samples	Average of formed inhibition zone (mm)	
	<i>E. coli</i>	<i>S. aureus</i>
A	7	10
B	8	12
C	12	15
D	9	9
E	8	8
F	0	0

### Instruments

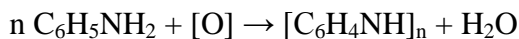
The UV-Vis spectrum was recorded by Shimadzu UV-vis spectrophotometer. A sonicator (Branson-1510) was used to obtain ultrasound of 40 kHz. Centrifuge machine (REMI) was used for centrifugation. Particle size and its distribution were recorded using Transmission Electron Microscope (TEM) (JEM-2100, 200kv, Jeol).

### Synthesis of Polyaniline

Although the synthetic methods to produce polyaniline are quite simple, the mechanism of polymerization is probably complex.

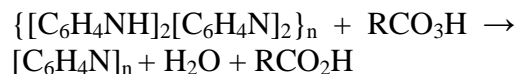
A. The formation of leucoemeraldine can be described as follows, where [O] is a generic oxidant (40):





The most common oxidant is ammonium persulfate. The components are each dissolved in 1 M hydrochloric acid (other acids can be used), and the two solutions slowly combined. The reaction is very exothermic. The polymer precipitates as an unstable dispersion with micrometer-scale particulates.

B. (Per) nigraniline is prepared by oxidation of the emeraldine base, one typical oxidant being meta-chloroperoxybenzoic acid (41):



C. Aniline hydrochloride and Ammonium Persulfate reacts to form Emeraldine (Fig. 12).

### **Synthesis of polyaniline Nanoparticles (Using sonochemical techniques)**

High-power ultrasound can generate cavitation within a liquid and through cavitation provide a source of energy which can be used to enhance a wide range of chemical processes. Such uses of ultrasound have been grouped under the general name sonochemistry. Ultrasound is defined as sound of a frequency beyond that to which the human ear can respond. The normal range of hearing is between 16 Hz and about 18 kHz and ultrasound is generally considered to lie between 20 kHz to beyond 100 MHz. Sonochemistry generally uses frequencies between 20 and 40 kHz. However since cavitation can be generated well above these frequencies, recent researches into sonochemistry use a much broader range (Fig. 12). High frequency ultrasound from around 5 MHz and above does not produce cavitation and this is the frequency range used in

medical imaging.

For the synthesis of nanostructured PANI sonochemical process is initiated with the drop-wise addition of an acidic APS (Ammonium persulfate) solution to an acidic aniline solution, similar to that of conventional PANI synthesis. However, the polymerization is accomplished with the aid of ultrasonic irradiation. Jing *et al.*, synthesized PANI nanofibers with high polymer yields using this technique 42-43. If the concentration of aniline and APS is high, three possible competitive reactions may be operative within the system: (i) the continuous formation of primary PANI nanofibers; (ii) the conversion of the primary nanofibers into thicker fibers with uneven surfaces; and (iii) the growth and agglomeration of the thicker fibers into irregular particles. In the conventional method of preparation, irregular PANI particles are obtained because of the simultaneous occurrence of reactions (ii) and (iii). While during the sonochemical synthesis, the further growth and agglomeration of the primary nanofibers are effectively prevented (even if more aniline and APS were added into the system), following the formation of more primary PANI nanofibers (42). One of the advantages of this approach is its scalability in comparison with other approaches such as interfacial polymerization or rapid mixing reaction.

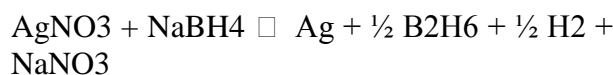
### **Synthesis of Polyaniline Composite**

With exposure to Ag nanoparticles on PANI/PVA composite, a new nanocomposite was obtained. The PANI/PVA/Ag nanocomposites were synthesized by chemical oxidation polymerization of aniline monomer in the presence of PVA and Ag nanoparticle colloidal solution. In a typical synthesis process aniline hydrochloride was added to the prepared Ag nanoparticles

colloidal solution (200ml). The obtained mixture was stirred for 10 min and then the PVA aqueous solution (which dissolve with 0.1M HCl) was added and the mixture was stirred for 30 min. By addition of the aqueous solution of ammonium persulfate, the mixture was allowed to react for 12h under constant stirring at -30C.

Metal nanoparticles are generally obtained from noble metals like silver, gold, platinum, titanium, copper and tin. Silver Nanoparticle colloidal solution was synthesized using NaBH<sub>4</sub> as a reducing agent. The chemical reduction of Ag<sup>+</sup> ions to Ag nanoparticles by

NaBH<sub>4</sub> is as follows:



The most possible approach to stabilise nanoparticles, especially Ag nanoparticles, and preparation of nanocomposites, is using polymers to improve polymer properties is to combine the desired polymer with other polymers which have better properties.

PANI has many advantages but has low solubility in several common solvents. IN fact, PVA improved the solubility of PANI and this novel composite can be applied for Ag nanoparticle coating.

The process of synthesizing PVA/PANI/Ag nanoparticles is shown in fig 13.

### **Characterization of synthesised Polyaniline Nanoparticles**

The TEM images of PANI nanofibers synthesized by the sonochemical method at different APS to aniline molar ratios are presented in Fig. 14. The diameter of the sonochemical synthesized PANI nanofibers with different APS to aniline ratios is ~50 nm.

However, the length of the nanofibers decreases with the increase in the APS to aniline ratio<sup>44</sup>.

### **Biological Relevance: Antibacterial property**

The antibacterial activity of the obtained nanocomposite was evaluated against Gram positive bacteria *Staphylococcus aureus* (*Staph. aureus*) and gram negative bacteria *Escherichia coli* (*E. coli*) using paper disc diffusion method. The antibacterial study showed that the PANI/PVA composite did not have a very good antibacterial activity but PANI/PVA/Ag composites were found to be effective against two bacteria. PVA/PANI/Ag nanocomposites were tested for antibacterial activity using *E.coli* and *Staph. aureus*. Fig. 15 shows the inhibition zones that were formed by nanocomposite samples. The diameter of the inhibition zones are 7, 8, 12, 9 and 8 mm and 10, 12, 15, 9, and 8 mm against *E. coli* and *Staph. aureus* respectively. The results are summarized and presented in Table 1. It is observed that PVA/PANI composite (sample of F) which was used as a control matrix, exhibited no antibacterial activity when compared with PVA/PANI/Ag nanocomposites. The scatter plot of the inhibition zone versus various PVA/PANI/Ag nanocomposites is presented in Fig. 16. According to Fig. 16 the PVA/PANI/Ag nanocomposite (C) with 15% Ag nanoparticle showed better antibacterial activity against *E. coli* and *Staph. aureus*. Silver exhibits outstanding antibacterial property that would lead to biomedical applications. The antibacterial activity of silver is dependent on Ag<sup>+</sup> that binds strongly to electron donor groups on biological molecules like sulfur, oxygen or nitrogen. The silver ions act by displacing other essential metal ions such as Ca<sup>2+</sup> or Zn<sup>2+</sup>. At low concentrations of nanoparticles, the interaction of particles with the cell wall of bacteria decreases and at the

high concentrations of the particles, the aggregation probability of particles increases, as a result, the effective surface to volume ratio of particles and so the resulting interaction between particles and the cell wall of bacteria decrease.

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