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Preparation, Characterization and Antimicrobial Properties of Silica Based Nanocoatings

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Abstract

In this paper organic-inorganic hybrid coatings with organic matrix (water soluble) containing SiNPs are synthesized. Stober process was used to prepare monodispersed silica nanoparticles. The reactions were carried out at high concentrations of sodium silicate [Na_2SiO_3]=0.1-1 M, low concentrations of ammonium hydroxide [NH_4OH]=, acetonitrile, in alcoholic mixture (ethanol, methanol and polyethylene glycol). The composition and morphology of coating formulations were estimated by infrared spectroscopy (FT–IR) and dynamic light scattering (DLS). The antimicrobial activities of these coating formulations were investigated against several pathogenic bacteria (*Streptomycin, Burkholderia pseudomallei, Salmonella gallinarum, Klebsiella pneumonia, Xanthomonas campestris and Pseudomonas fluorescens*). It was revealed that resulting formulations containing SiNPs persist their antimicrobial effect over reasonable storage time.

Keywords: Infrared spectroscopy; Organic matrix; Conductivity; Hydrolysis

Introduction

Since last few decades nanotechnology is considered as major area of research in field of science and technology e.g., space science, medical, coatings and electronics [1]. It is the study and use of structures which have less than 100 nanometers (nm) size [2]. Nanotechnology promises to unleash vast potential in the field of coatings. World widely considerable exertion on nanoscale coatings is being in progress. The paint and coating industries considers the use of nanoparticles as fillers in coatings with modified surface properties [3]. Nanocoatings are produced by the incorporation of nanoparticles (1-100 nm) in coatings formulations that enhance specific features. Due to small size and large surface area of nanoparticles paint and coating industry get many advantages and opportunities [4]. Addition of nanoparticles to coatings improves coating's properties e.g., object's appearance, self-cleaning power, corrosion resistance, moisture absorbance, conductivity and optical properties [5] and help in producing multipurpose coatings with a little cost difference. Transparency, flexibility, gloss and strength of coatings changed according to composition of nanoparticles [6]. The concept of increasing coating's biocidal activity with using inorganic NP is being developing [7]. Nanocoatings can be applied in paints, textiles, plastics and minerals etc.

Silica nanoparticles are very important in coatings because they have low toxic level, long durability and are flexible to functionalize with versatile molecules and polymers [8]. Reported harmless exposure level of SiNPs (19 nm) is 243 µg/L for 48 hours while for AgNPS (25 nm) is 27 µg/L for 48 hours [9]. in this paper we studied the acid catalyzed preparation of SiNPs from sodium silicate and their biocidal effects in coatings (Figure 1).

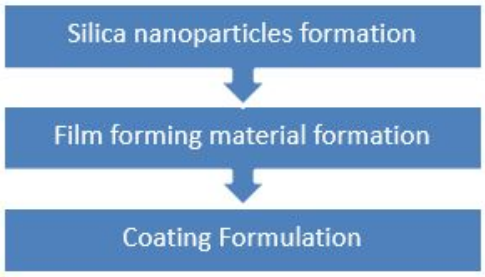

Figure 1: Silica nanocoatings-formation.

Materials and Methods

Reagents

Yeast, Agar-Agar (Chaitanya Biological Pvt. Ltd.) and Trypton (Alliance Pvt. Ltd) are used for antibacterial analysis. Ethanol (99.9%) and methanol (99.9%) are purchased from Merck Darmstadt., Germany. Acetonitrile (99.9%) is used as catalyst for hydrolysis and condensation of sodium silicate (Na_2SiO_3), both of are lab products. Tween 80, Calcium carbonate and barium sulphate are purchased from Sigma, Fisher Scientific U.K Limited Bishop Meadow Road and May and Baker Ltd. Dagenham, England respectively. The following densities and molecular weights are used estimate the concentrations of various reagents: MW sodium silicate=122.06 g/mol, MW water=18 g/mol, MW $(NH_4)2PO_4$=149.09 g/mol.

Experimental procedure

The most important factors involved in the production of silica based antimicrobial nanocoatings are: stability, adhesion and good dispersion of silica nanoparticles in organic matrix. For coating formulations SiNPs with six different compositions were prepared by Stober process in which sodium silicate was used as precursor of silica [10]. Stober process mainly involves the hydrolysis and condensation of alkyl silicates. First a certain amount of deionized water, acetonitrile, ammonium phosphate and alcohol mixture (ethanol and methanol 50, 50%) were blended for 10 minutes in beaker. Then 5 ml sodium silicate (0.1-1 M) was added drop wise in the above solution (Table 1).

Sample No.	Na_2SiO3 (M)	H_2O (ml)	Alcohol (ml)	Acetonitrile (M)	$(NH_4)2PO_4$ (M)	Temperature (°C)	Duration (min)
A	0.1	1	10	0.1	0.1	20	5
B	0.2	2	15	0.2	0.2	30	5
C	0.4	3	20	0.3	0.3	22	5
D	0.5	4	25	0.4	0.4	60	4
E	0.7	5	30	0.5	0.5	5	3
F	1	6	35	0.6	0.6	5	2

Table 1: SiNPs preparation in different compositions.

During continuously magnetic stirring (1-5 hours) at 5-20°C. SiNPs formed in the form of clear solution. This method results in the formation of 1-100 nm silica nanoparticles. In second step film forming material was prepared by mixing certain amount of distilled water, Calcium carbonate, barium sulphate, tween80 (surfactant) and sodium hydroxide solution, this mixture was mixed for 20 minutes with magnetic stirring, white milky solution appears [11]. Then trimethylchlorosilane (TMCSi) as coupling agent was added in variable amount during magnetic stirring (for 10 minutes) at last SiNPs of different compositions were added with different concentrations to formulate six different types of nanocoatings, and this mixture was continuously stirrered for 15-20 minutes for efficient mixing of inorganic particles in organic matrix (Table 2).

Sample no.	H_2O (ml)	$CaCO_3$ (G)	$BaSO_4$ (g)	Tween80 (ml)	NaOH (ml)	SiNPs (ml)	TMCSi (g)
A	5	0.5	0.5	5	5	15	2
B	10	1	1	5	5	15	2
C	5	1.5	1.5	5	5	15	2
D	5	2	2	5	5	15	2
E	5	2.5	2.5	5	5	15	2
F	5	3	3	5	5	15	2

Table 2: Coating formulations with SiNPs.

Conductivity measurement

Conductivity of silica nanocoatings get enhanced as the amount of adsorbed water increased. This enhancement is due to the cationic conduction, as the silanol groups are surrounded by hydroxyl groups the number of mobile protons increases because the polar hydroxyl groups increase the dielectric constant of silica coatings and decreases the dissociation energy of protons [12]. Electrical conductivity of prepared nanocoatings was determined by using conductometer (Ec 215 HANNA instrument).

pH measurement

pH of the coatings has very significance in their biocidal action. It also relates with the conductivity. As the conductance increases due to free hydroxyl protons pH decreases. This acidic nature is helpful in enhanced microbial resistance. pH of the nanocoating formulations was measured with potentiometer (AD1030, HANNA instrument).

Structural analysis

Foriour transform infrared spectroscopy (FTIR) is a technique to analyze the different functional groups present in Chemicals. Nanosamples were analyzed by putting their drops on KBr pellets in sandwich form under 20 N/m^2 pressure. IR spectrum of all nanocoatings was recorded by using (Jasco FT/IR-4100-type A, serial number B032961016) instrument.

Particles size determination

Dynamic light scattering, DLS method is very popular to determine the size of small particles which are in Brownian motion in a solution. Using this method, a commercial dynamic light scattering instrument was used and size of the SiNPs particles was determined.

Antibacterial activity test

Six different pathogenic bacteria species were collected from fungal culture bank (FCBP) from institute of agriculture sciences university of Punjab Lahore, Pakistan. LBA (Luria-Bertani-Agar) medium was used for antibacterial assay. Pathogenic strains which were to be analyzed against nanoformulations were sub cultured on LBA medium in autoclaved (Tomy, SX-700, instrument) petri dishes in incubator (Memmert) for one day at 37°C. Well method was used for this analysis. Inhibition zones for all nanosamples were measured in terms of centimeter (cm) [13]. To avoid any contamination most of the test

was performed under laminar flow cabinet (EN 1822, class H13 HEPA Filters, US Federal standard 209E class 10, Whitehouse Singapore).

Results and Discussion

Conductivity measurements

Electrical Conductivity of prepared nanocoatings was measured at regular intervals. As the amount of added buffer Solution (water) increased, conductivity also showed increase as shown in Figures 2-4.

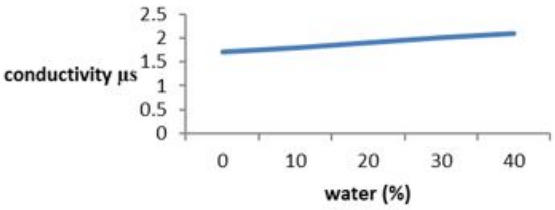

Figure 2: Electrical conductivity of nanocoatings.

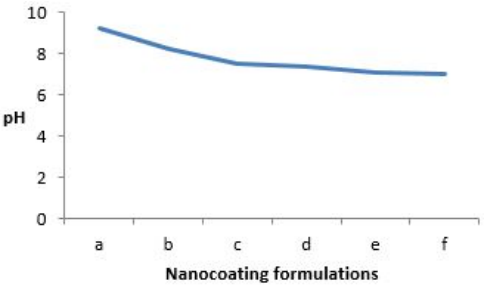

Figure 3: Due to increased water concentration the hydroxyl groups polarized the silanol groups and release the free protons which cause decrease in pH from formulation A to F. Stability conformation.

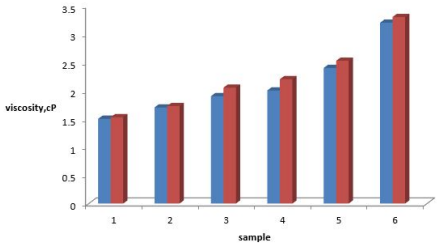

Figure 4: Viscosity stabilities immediately after formation at 20°C (column 1-blue) and after six months storage at 45°C (Column 2-red).

FTIR analysis

FTIR analysis of the prepared samples was performed and obtained spectrums showed a regular pattern for all nano samples as shown in Figure 5.

Particle size measurement

Here, particle size was determined by two methods which is shown in below Figures 6A and 6B.

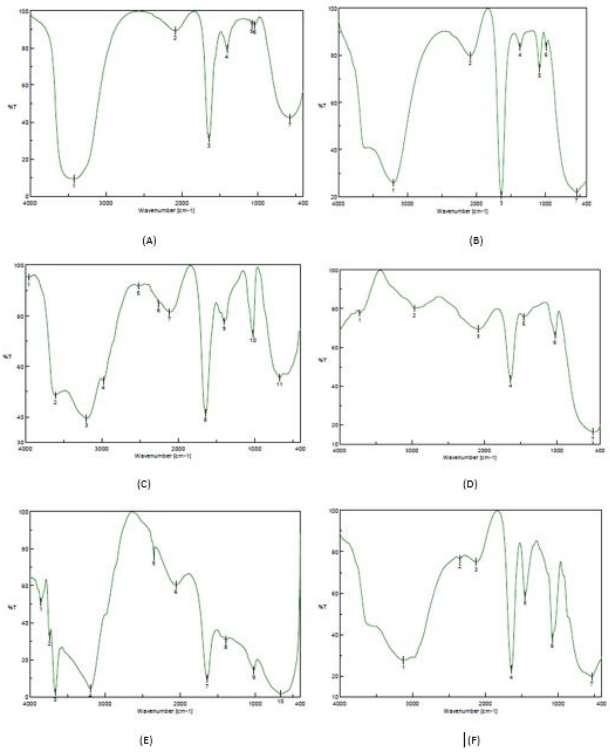

Figure 5: A-F. Indicates the I.R spectra's of sample a, b, c, d, e and f. In all spectra's the broad and intense peaks in the wave number range of 3242.96 cm^{-1} to 3900 cm^{-1} alcoholic group in stretching mode. The band at 1080 cm^{-1}-1100 cm^{-1} is assigned as asymmetric stretching vibrations of Si-O-Si which indicates the formation of silica network. 2056.71 cm^{-1}-2750 cm^{-1} absorbance represents the CO group with medium intensity and stretching vibrations. this suggest that there are no significant changes in structures of nanoformulations A, B, C, D, E and F.

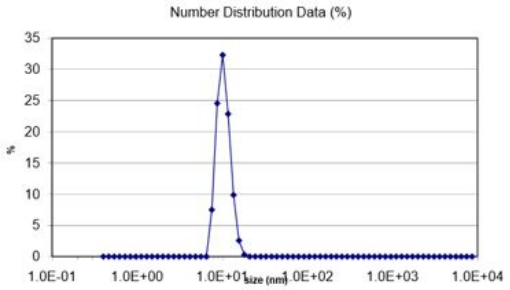

Figure 6A: Particle size was determined by light scattering method. Peak shows 1.2 nm particle size with 32.5% intensity.

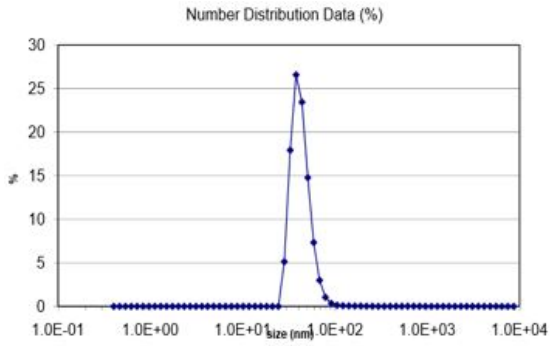

Figure 6B: Size measurement by Dls. Peak shows 11 nm size with 27% intensity. Precursor effect on particle size

As the concentration of sodium silicate increase the diameter of SiNPs increased, (reported by Xiao) and has been confirmed by our experimental results as shown in Figure 7.

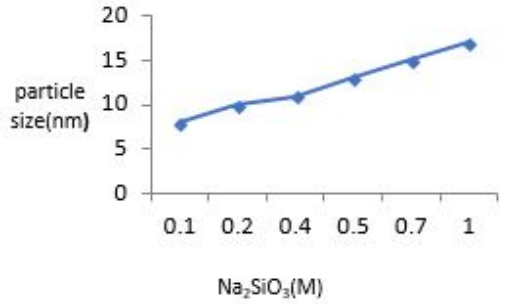

Figure 7: Precursor effect on particle size.

Antibacterial analysis

Silicananoparticles kills the bacteria by attachment with outer wall. Nanoparticles interacts the wall by hydrogen bonds between silanol groups and bacterial wall's functional groups. These bonds destabilize the peptidoglycan (bacterial wall) and kill the bacteria [14].

The bacterial resistance of antimicrobial coatings was determined with Agar well method. The nanocoating formulations were tested against six pathogenic strains of microorganisms: *Streptomycin, Burkholderia pseudomallei, Salmonella gallinarum, Klebsiella pneumonia, Xanthomonas campestris* and *Pseudomonas fluorescens*. The tests were conducted in parallel with control bacteria. The results are shown in Figures 8 and 9.

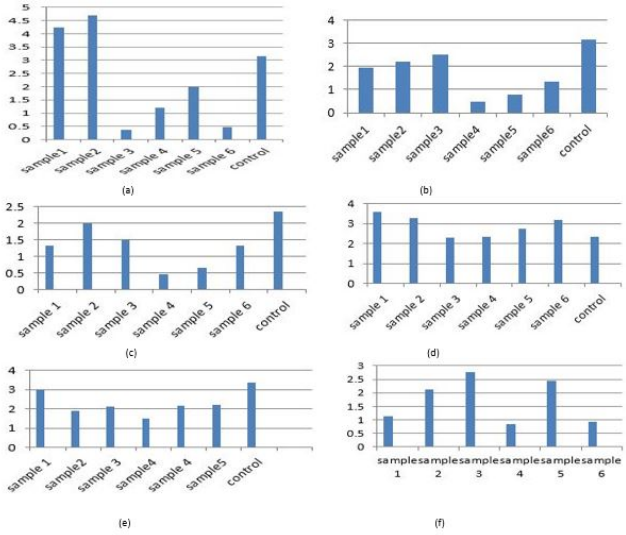

Figure 8: A-F. These figures indicates that all coatings have antibacterial activity in between 80% to 99%. Thier bacterial resistance power is variable according to composition. Sample A shows the best inhibizition zones (IZ) with *Pseudomonas fluorescens* close to control bacteria. Least growth zone is developed by sample C against *Pseudomonas fluorescens* and *Salmonella gallinarum* while it represents the utmost inhibition zone with *Xanthomonas campestris*. The SiNPs content have positive influence on antimicrobial activity i.e., sample with improved SiNPs concentration has large bacterial growth prevention zone e.g., sample A (20 ml) and C (15 ml).

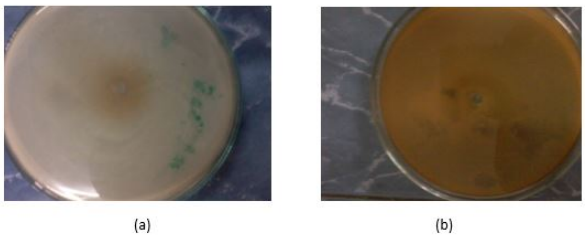

Figure 9: Representative images for agar well diffusion method employed to determine antibacterial activity of nanoformulations.

Conclusions

A comprehensive method was developed to formulate organic-inorganic hybrid coatings by SiNPs incorporation, with use of low energy and simple heating. It was shown that our all formulations have antimicrobial activity against *Streptomycin, Burkholderia pseudomallei, Salmonella gallinarum, Klebsiella pneumonia, Xanthomonas campestris* and *Pseudomonas fluorescens* pathogenic bacteria species. SiNPs was shown to reserve their structural properties after long time storage with FTIR.

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