CHAPTER 4
SYNTHESIS, CHARACTERIZATION AND MICROENCAPSULATION PROCESS OF THE NANO SILICA

4.1 INTRODUCTION

This chapter is concerned with the synthesis of nano silica particles from the natural resources such as rice husk. The synthesized nano silica particles were characterized by X-ray diffractometer (XRD), Scanning Electron Microscopy (SEM) and High Resolution Transmission Electron Microscopy (HR-TEM). This chapter also deals with the preparation and characterization of nano silica encapsulated microcapsules.

4.2 SYNTHESIS OF NANO SILICA PARTICLES

Natural materials such as rice husk and sugar cane waste are rich in silica. The yield obtained from sugar cane is very low (around 2%), but the rice hull gives around 30%. In the present work, amorphous nano SiO₂ particles were synthesized from the rice hulls by Thermal Degradation Method. The process flow chart for the synthesis of nano silica particles is given in Figure 4.1.
Figure 4.1 Process flow chart for synthesis of nano silica

Rice hulls → Rinse with distilled water → Dried under room temperature → Again dried under 80°C for 1 hour → Grinded and the hulls are converted to micro particles → The powder is refluxed with 10% HCl for 30 min → The residue is dried at 80°C for 30 min → The residue is again kept under muffle furnace at 750°C for 3 hrs → Nano silica powder → Grinding the powder by ball milling machine to get uniform particles → Conversion of microencapsulated nano silica
4.3 CHARACTERIZATION OF NANO PARTICLES

The synthesized nano silica particles were characterized by X-Ray Diffractometer to find out the nature of the particles whether it is amorphous or crystalline. Scanning Electron Microscope (SEM) and High Resolution Transmission Electron Microscope (HRTEM) were used to find out the size of the nano silica particles. Optical microscope was used to characterize the microcapsules.

4.3.1 X-Ray Diffraction (XRD) Pattern of Nano Silica

The synthesized nano silica particles were characterized under X-Ray Diffractometer (XRD) using CuKα (λ = 1.54Å) as a radiation source. The XRD patterns of the nano silica powder obtained from rice hulls is shown in Figure 4.2. The powder diffraction pattern indicates a broad peak at 2θ = 22°, which reveals the amorphous nature of the nano silica. Further, the XRD pattern confirms the absence of any ordered crystalline structure.

![XRD pattern of nano silica](image)

Figure 4.2 XRD pattern of nano silica
4.3.2 SEM and HRTEM Analysis of Nano Silica

The synthesized particles are white in colour. The particle size was analysed by Scanning Electron Microscope (SEM) and shown in Figures 4.3 and 4.4. The image shows the agglomerated silica particles. High Resolution Transmission Electron Microscope (HRTEM) was used to measure the particle size and the image of nano particle was shown in Figures 4.5 and 4.6. It shows that the particles are porous in nature, nano meter range and the particle sizes vary from 50 to 100 nm.

4.4 PREPARATION OF MICROCAPSULES

Microcapsules would be a system that is easy to apply, does not affect the existing textile properties and has increases the life of a garment that allows normal fabric-care processes to take place. Currently, although capsules can survive 25 to 30 wash cycles, conventional ironing and other heat-input processes such as tumble-drying can cause a dramatic reduction in the desired effect. The microencapsulation is one of the latest techniques used to apply nano metal oxide particles. Sodium alginate and gelatin are the widely used wall materials.

In this process, Sodium alginate (NaC\textsubscript{6}H\textsubscript{7}O\textsubscript{6}) and glycerol (99.9 %) were used for the microencapsulation process with nano silica as a core material. The aqueous suspension of different concentration of (1%, 1.5% and 2%) nano silica particles were prepared under sonication for 5 minutes. The highly dispersed solution of nano silica (aqueous suspension of nano silica) was added drop-by-drop in to sodium alginate solution under stirring at a constant temperature of 50°C. The pH value of the solution was adjusted to 4 by adding acetic acid with continuous stirring for 30 min and the temperature of the solution was reduced below 30°C till the formation of microcapsules was observed. The wall of the microcapsules was stabilised by bringing the pH value as 4 and the addition of glycerol as a hardening agent.
Figure 4.3 SEM image of agglomerated nano silica particles in 2 µm units

Figure 4.4 SEM image of agglomerated nano silica particles in 5 µm units

Figure 4.5 HR TEM image of nano silica particles in 100 nm units

Figure 4.6 HR TEM image of nano silica particles in 50 nm units
4.5 CHARACTERIZATION OF MICRO CAPSULES

The high resolution optical microscope was used to characterize the microcapsules. The image of the microcapsules is shown in Figure 4.7. It is evident from the image that the formation of the microcapsules obtained employing the simple coacervation process. Figure 4.8 shows the transparent microcapsules containing only sodium alginate shell material, while the black circle indicates the encapsulation of core nano silica particles. The produced microcapsules were in small spherical shape and the average microcapsule size ranges from 25 μm to 40 μm.

![Image of Sodium alginate with nano silica encapsulated Microcapsules](image1)

![Image of Sodium alginate without nano silica encapsulated Microcapsules](image2)
4.6 CHARACTERIZATION OF NANO SILICA COATED FABRICS

Figure 4.9 show that the SEM image of microencapsulated nano silica coated cotton fabric. This image confirms the presence of microencapsulated nano silica on the surface of cotton fibres.

Figure 4.9 SEM image of microencapsulated nano silica coated cotton fabric