CHAPTER 2
EXPERIMENTAL METHODS AND MATERIALS
Chapter 2 Experimental Methods and Materials

2.1 Materials

The chemicals and solvents used for the present study were procured from nice chemical Pvt, E. Merck, spectro chem. and himedia chemicals.

2.1.1 Chemicals

The common names and grades of supply are given as below.

<table>
<thead>
<tr>
<th>S.N.</th>
<th>Chemicals</th>
<th>Grade</th>
<th>Source of Supply</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Silicon oil</td>
<td>Laboratory reagent</td>
<td>Nice Chemicals Pvt. Cochin</td>
</tr>
<tr>
<td>2</td>
<td>Acrylic acid</td>
<td>Laboratory reagent</td>
<td>Central Drug House Pvt. Limited</td>
</tr>
<tr>
<td>3</td>
<td>Metha acrylic acid</td>
<td>Laboratory reagent</td>
<td>Spectro Chem. India</td>
</tr>
<tr>
<td>4</td>
<td>2,2 Azobisisobutyro nitrite</td>
<td>Analytical reagent</td>
<td>Ms. Himedia chemicals</td>
</tr>
<tr>
<td>5</td>
<td>Methanol</td>
<td>Analytical reagent</td>
<td>Glaxo laboratory (India) Ltd.</td>
</tr>
<tr>
<td>6</td>
<td>Oleic acid</td>
<td>Analytical reagent</td>
<td>Moly chem.. Mumbai</td>
</tr>
<tr>
<td>7</td>
<td>Ferrous chloride</td>
<td>Extra pure</td>
<td>Moly chem.. Mumbai</td>
</tr>
<tr>
<td>8</td>
<td>Ammonium hydroxide</td>
<td>Extra pure Sp. Gr. 0.89</td>
<td>LOBA CHEMIE Pvt. Ltd. Mumbai</td>
</tr>
</tbody>
</table>
2.1.2 Water

Deionised water was distilled twice with a small quantity of alkaline potassium permanganate. Then, water was distilled in a quick fit apparatus with sulphuric acid. The specific conductance of the distilled water prepared for the present study was in the order of < 2×10⁻⁶ Ω cm⁻¹ [1].

2.1.3 Preparation of ferrous chloride solution

The solution of ferrous chloride [FeCl₂·4H₂O] was prepared by dissolving 3.0gm of sample in 100ml deionised water in order to obtain the solution strength of 0.15M. This solution was always prepared fresh before use.

2.1.4 Ammonium hydroxide

The solution of ammonium hydroxide was prepared by calculated dilution of concentrated reagent obtained from the source. For the preparation of the solution the density value given on the level was used and the following relation was neutralized.

\[
\frac{\text{Molecular weight} \times V_1}{\text{Specific gravity}} = M_2N_2
\]

Molecular weight of NH₄OH = 35.5

\[
\frac{35.5 \times V_1}{0.91} = 7 \times 250M
\]

\[
V_1 = 44.859 \text{ ml}
\]
The 44.859 ml of NH₄OH were diluted to the final volume 250 ml in a measuring flask of corning glass. The strength of this solution was checked by titrating it against standard hydrochloric acid using methyl orange indicator.

2.1.5 Silicon oil

Silicon oil is a synthetic chemical which is also known as silicone. It has a wide application in industries and laboratories due to its characteristics. The specific properties of this oil is chemical stability, thermal stability, non-toxicity, environmental capability, fire resistance and non reactive with most of the chemicals and substrates. Its non-flammable property is also important from safety point. It has antifoaming property which is also useful for detergent industries.

The silicon oil can be obtained by combining silicon natural element with carbon, hydrogen, oxygen and various other chemical elements to get the product of desired structure. In this study we have used poly-di-methylsiloxane (PDMS) as silicon oil.

2.1.5.1 Synthesis of Poly-di-methylsiloxane

The chemical formula for PDMS is CH₃[Si(CH₃)₂O]ₙSi(CH₃)₃, where n is the number of repeating monomer [SiO(CH₃)₂] units.

Industrial synthesis can begin from di-methylchlorosilane and water by the following net reaction.

\[
n\text{Si(CH₃)₂Cl₂} + n\text{H₂O} \rightarrow [\text{Si(CH₃)₂O}]_n + 2n\text{HCl}
\]

(1.15)
The structure of PDMS is given below.

![PDMS Structure](image)

The polymer is manufactured in multiple viscosities, ranging from a thin pourable liquid (where \( n \) is very low), to a thick rubbery semi-solid (where \( n \) is very high). PDMS molecules have quite flexible polymer backbones (chains) due to their siloxane linkages, which are analogous to the ether linkages used to impart rubberiness to polyurethanes. PDMS have unusually high level of visco-elasticity. PDMS is visco-elastic, meaning that at long flow times (or high temperatures), it acts like a viscous liquid, similar to honey. The polymerization of PDMS depends upon the second constituents in contact and related work has been carried out in recent past [2].

### 2.1.6 Acrylic acid

Acrylic acid has two reaction points or functional groups which facilitates the polymerization under suitable conditions. It is unsaturated carboxylic acid, having a vinyl group directly connected to carboxylic acid terminus. It is also known as prop-2-enoic acid. It can be obtained from propene a byproduct of ethylene and gasoline product.
The polymerization may take place to form polyacrylic acid or other monomeric form having completely different physical and chemical properties than their monomers.

The structural formula of the acrylic acid is given below.

\[
\text{CH}_2\text{CCH}_2 + 1.5 \text{O}_2 \rightarrow \text{CH}_2=\text{CHCOOH} + \text{H}_2\text{O}
\]

\[\text{(1.16)}\]

2.1.7 Methacrylic acid

Methacrylic acid is also known as 2-methyl propenoic acid. In comparison to acrylic acid it polymerizes very readily in glacial form. It has terminals for polymerization process and can be used in textile, leather and other surfaces in the industries. It has solubility in warm water and miscibility with most organic solvents. The structural formula of the acid is given below.
2.1.8 Oleic acid

The source of oleic acid is vegetable fats, oils and various animals. It is natural fatty acid. It is classified as mono-unsaturated omega-9 fatty acid. The term oleic means related to or obtained from oil or olive. In the literature [3] the formula of the oleic acid has been reported as \( \text{CH}_3(\text{CH}_2)_{7}\text{CH}=\text{CH}(\text{CH}_2)_{7}\text{COOH} \). The structural can be illustrated as below.

In this study the oleic acid has been used as stabilizing agent for the nanoparticles obtained from ferrite and grafts. It has emulsifying or solubilizing properties. It has tendency to undergo the reactions of carboxylic acids and alkenes both. In aqueous base solution it can give soaps known as oleates.
2.1.9 2,2- Azobisisobutyronitrile [AIBN]

Azobisisobutyronitrile is commonly known as AIBN. It is soluble in ethanol and other organic solvents but it is insoluble in water. On decomposition it forms two-2-cyanoprop-2-yl-radicals and nitrogen gas. This characteristic of compound is useful to initiate free radical polymerization and other radical reactions. The decomposition reaction is given below.

![Structure of AIBN]

2.2 Characterization

The characterization of the nanomaterials was carried out in the following institutions.

(i) Department of chemistry G.B.Pant University of Agriculture and Technology Pantnagar Uttarakhand

(ii) Sophisticated Analytical Instrument Facility, Central Drug Research Institute (SAIF) Lucknow.

(iii) Institute instrumentation center, Indian Institute of Technology Roorkee Uttarakhand.

(iv) All India Research Facility (AIRF) Jawaharlal Nehru University New Delhi.

The tests of various instruments used in the study are listed below with their models.
(i) XRD, model-RINT/DMAX 2200 H/PC (Rigaku, Japan)
(ii) FT-IR, model- Galaxy 300 Mattson.
(iii) VSM, model- Princeton EG & G Applied Research VSM, 155.
(iv) TG-DTA-DTG, model-EXSTAR TG/DTA 6300.
(v) \(^1\)H-NMR, model- Brucker Av 400.
(vi) UV-visible, model-Genesis 10 Thermospectronic USA.
(vii) Ultra-Sonation, model PCI 3.5L.

References
1. P.S.Nikam and A.B.Sawant, Viscosity of potassium halides and symmetrical tetraalkylammonium bromides in acetonitrile + water mixtures at 298.15K.


