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An analysis of cetylpyridinium chloride (CPC) by FESEM, EDX methods, and its applications

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Abstract

Cetylpyridinium chloride (CPC) is a potential cationic surfactant with cleansing and antimicrobial properties. Apart from its use as an emulsifying and cleansing agent, it is used in cosmetics, industrial processes, pharmaceuticals, food additives, etc. In the present study, the purity of cetylpyridinium chloride (CPC) was determined using FE-SEM and EDX methods. The FE-SEM test showed CPC sizes from 46.35µm to 77.76µm. The result and analysis of EDX depicted the purity of the sample. The percentage of elements present in cetylpyridinium chloride has been explained by EDX data. The percentages of C, N, and Cl were found to be 87.4, 5.4, and 7.2 percent respectively by weight.

Keywords: CPC, Surfactant, Antimicrobial, FE-SEM, EDX

1.0 Introduction

Cetylpyridinium chloride (CPC) with IUPAC name pyridinium, 1-hexadecyl chloride is a quaternary ammonium compound that is commonly used as an antimicrobial and antiseptic agent (Rafati et al., 2008). It is prepared by the reaction between cetyl alcohol with pyridine in the presence of HCl. It has a broad range of applications in different oral care products like mouthwashes, lozenges, and toothpaste. It is also used in the treatment of oral infections, gingivitis, and halitosis. CPC is a white powder having molecular formula $C_{21}H_{38}ClN$, and having solubility in water and alcohol. It is amphiphilic as it contains a cetyl group ($C_{16}H_{33}$) or hexadecyl group containing 16 carbon alkyl chain which acts as a hydrophobic or lipophilic part (Shahi et al., 2023a). A CPC molecule contains a positively charged pyridinium group as the hydrophilic part which contains a positively charged nitrogen atom in the pyridinium ring (Yadav et al., 2024a). The negatively charged chloride (Cl^-) ion is linked with the positive charge to balance the charge. It is a highly efficient noble compound with both surfactant properties and microbial activity.

CPC is a cationic surfactant having a positive charge in its hydrophilic or polar head. The molecular structure of CPC with its hydrophobic part and hydrophilic part is represented in Fig. 1. The detergency activity of CPC is due to its amphiphilic nature, i.e. due to the presence of hydrophilic or polar head and hydrophobic or non-polar tail in the same molecule. The positively charged pyridinium ring which is the main constituent of its hydrophilic head interacts with water. Similarly, the cetyl group, or the long carbon chain composed of 16 carbon atoms acts as a hydrophobic tail that interacts with lipid, oil, and debris. It reduces the interfacial surface tension and spreads easily. It readily emulsifies oil, dirt, and other organic matter in water and helps in their effective removal (Ali et al., 2014).

CPC is chemically stable, maintaining its efficacy over a wide range of conditions, which contributes to long-lasting antimicrobial protection in products. The advantages of cetylpyridinium chloride (CPC) include its broad-spectrum antimicrobial effectiveness, safety, versatility, and ability to enhance oral hygiene and prevent infections (Mao et al., 2020). These attributes make it a valuable component in a variety of consumer, healthcare, and industrial products. An interesting investigation is to observe the physicochemical properties of CPC in water and mixed solvent media at different temperatures. There was a study of the effects of methanol, dimethyl sulfoxide, and temperature on the micellization of cetylpyridinium chloride (Bhattarai et al., 2017). In ethanol-water mixed solvents, cetylpyridinium chloride's critical micelle concentration depends on the solvent composition (Bhattarai et al., 2012). The interaction of salts with organic additives affects the micellar association of cetylpyridinium chloride (Kabir-ud-Din et al., 1997).

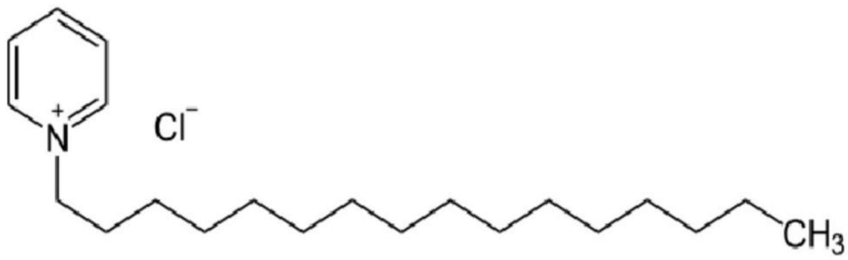

Figure 1: General representation of CPC molecule showing hydrophilic head (pyridinium cation $[C_5H_5NH]^+$) and hydrophobic tail or cetyl group ($C_{16}H_{33}$).

As corrosion inhibitors for mild steel in sulphuric acid solution, single surfactants such as CPC and mixed surfactants were studied (Shahi et al., 2024; Yadav et al., 2024b; Yadav et al., 2024c; Zhu et al., 2015). There are several studies about the interaction of the dye with cetylpyridinium chloride (Simončič et al., 2000; Shahi et al., 2023a; Shrestha et al., 2018; Shahi et al., 2023b). The interaction of dye and mixed surfactants is very popular nowadays in industrial areas (Kartal et al., 2005; Yadav et al., 2024a; Shah et al., 2022).

Polyelectrolyte-micelle complexes were examined for the effects of concentration and polymerization degree on the lifetime and association degree of dye molecules (Bulakov et al., 2007). They used cetylpyridinium chloride as the surfactant whereas polyacrylic acid (PAA) was the polyelectrolyte. The dye molecules as anionic dye eosin (E) were used as the luminescence probe. There were some studies of the solution properties of polyelectrolyte-surfactant and dye interactions (Yadav et al., 2022; Yadav et al., 2024d).

The field emission scanning electron microscope (FE-SEM) captures the microstructure of materials. Rather than using light sources, it uses electrons to study the topography of objects (Jaya, 2020). An energy dispersive X-ray analysis (EDX, EDS, or EDXA), also called energy dispersive X-ray spectroscopy, is a way to use X-rays to find out what elements are in something or to describe its chemical make-up (Gupta et al., 2020). FE-SEM images were taken of mild steel surfaces in 0.05 M H_2SO_4 with and without 0.0077 M CPC. When mild steel was immersed in 0.0077 M CPC inhibited in H_2SO_4 solution for 6 hours at 25 °C, EDX data indicated the presence of a nitrogen peak of the CPC (Yadav et al., 2024c). In this study, our objective is to observe the analysis of cetylpyridinium chloride (CPC) by FE-SEM and EDX. In addition, the study aims to see how CPC can be applied to a variety of research fields.

2.0 Materials and Methods

2.1 Materials

The material used in this study was cetylpyridinium chloride (CPC). It was purchased from HIMEDIA, India.

2.2 Methods

FE-SEM (model: JSM- IT800 Schottky Field Emission Scanning Electron microscope, Japan) and EDAX Octane (Model: Elect Super Energy Dispersive X-ray Spectroscopy, USA) were used for the characterization.

3.0 Results and Discussions

Field Emission Scanning Electron Microscopy (FE-SEM) is a powerful tool for characterizing the surface morphology and structural details of materials at the nanoscale. To produce electrons in FE-SEM, the filament is positioned in a strong electric potential gradient. Electrons are often produced using tungsten (W) wire. This method is often referred to as a cold cathode field emitter because it doesn't need heat. The sample lies at the focus of the electron beam, and the beam that is produced following interactions between the electron and the sample includes all of the data pertaining to the material's morphology (texture), crystalline structure, orientation, and chemical composition. It is among the most effective methods for determining the sample's composition, morphology, and surface structure. It offers a high-resolution, three-dimensional surface image of the substance. High resolution has the benefit of making it simple to identify closely spaced particles by increasing magnification (Kumar et al., 2023).

When analyzing cetylpyridinium chloride (CPC) using FE-SEM, we are typically interested in observing the morphology of CPC aggregates, crystallization patterns, or how CPC interacts with other materials in a given sample to analyze FE-SEM images of CPC. A quaternary ammonium compound may form crystalline structures. These can appear as sharp-edged platelets, rods, or layered structures depending on the crystallization environment. CPC also forms micelles or other surfactant aggregates, which may appear as clusters or spherical particles in FE-SEM images. The size and distribution of these aggregates can give insights into their self-assembly behavior. CPC aggregates or crystals are typically measured in the nanometer to micrometer range. FE-SEM software often has built-in tools for size measurement. The particles shape variation was examined, which could indicate different phases of crystallization or interaction with other compounds (e.g., polymers, proteins). The texture of the CPC crystals or aggregates was investigated. Smooth surfaces often indicate well-formed crystals, while rough or irregular surfaces might suggest incomplete crystallization or interactions with other compounds.

FE-SEM image could provide a thorough visual representation of the surface morphology of cetylpyridinium chloride (CPC) at the micro- or nanoscale. Cetylpyridinium chloride's FE-SEM image (Fig. 2) usually shows the compound's surface much magnified, making it possible to observe its structural characteristics, including molecular arrangement, crystalline structures, and any surface imperfections or textures. A focused electron beam would be used to scan the sample's surface, and the signals produced by the interactions between the electrons and the sample's atoms would be detected to create the image.

Important details on the morphology and structure of materials can be revealed by FE-SEM images, which is useful for a number of academic disciplines, including chemistry, materials science, and nanotechnology. Researchers could learn more about CPC's physical characteristics, behavior in various environments, and interactions with other substances by using these kinds of images. The FE-SEM test revealed CPC sizes ranging from 46.35 µm to 77.76 µm (Fig. 2). This data can be essential for basic study into the characteristics of CPC as well as for improving the performance and formulation of products containing this molecule

Energy Dispersive X-ray Spectroscopy (EDX or EDS) is commonly used alongside FE-SEM to determine the elemental composition of a sample. It is one of the common microanalytical methods for determining and measuring the elements in a sample. The sample is bombarded with a concentrated beam of high-energy electrons during EDS analysis. Some of the atoms' inner shell electrons are knocked out of their orbits when these electrons interact with the atoms in the sample, creating vacancies in the inner electron shells (Hodoroaba, 2020). However, when analyzing a chemical compound like cetylpyridinium chloride (CPC) using EDX, there are some important points to consider regarding the elements present and the limitations of the technique. CPC

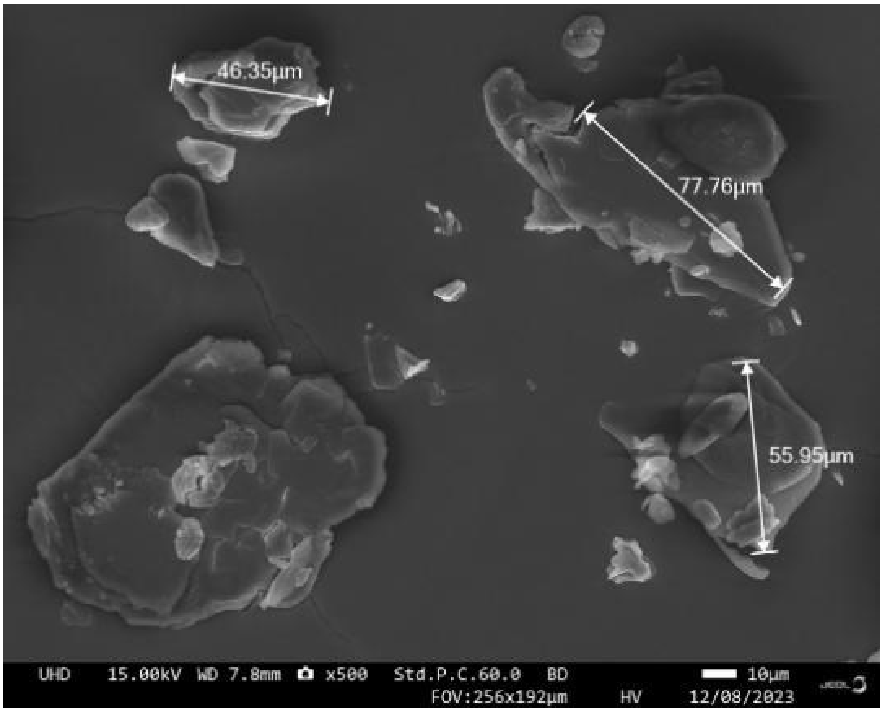

Figure 2: FESEM image of CPC

($C_{21}H_{38}ClN$) contains carbon, nitrogen, chlorine and hydrogen, however hydrogen cannot be detected by EDX.

First, the sample of CPC was dried in the oven at 50 ℃ for about 24 h. Then it was kept in the desiccator. After 7 days, it was tested using FE-SEM and EDX instruments by making fine powder of CPC. The EDX spectra showed peaks for carbon (C), nitrogen (N), and chlorine (Cl) as shown in Fig.3. Carbon peak will likely be the most dominant peak since CPC contains a long hydrophobic tail ($C_{21}H_{38}$). Nitrogen peak is present due to the quaternary ammonium group (-$CH_3N^+(C_6H_5)$) of CPC. Chlorine peak should appear distinctly, as CPC contains a chloride ion (Cl^-).

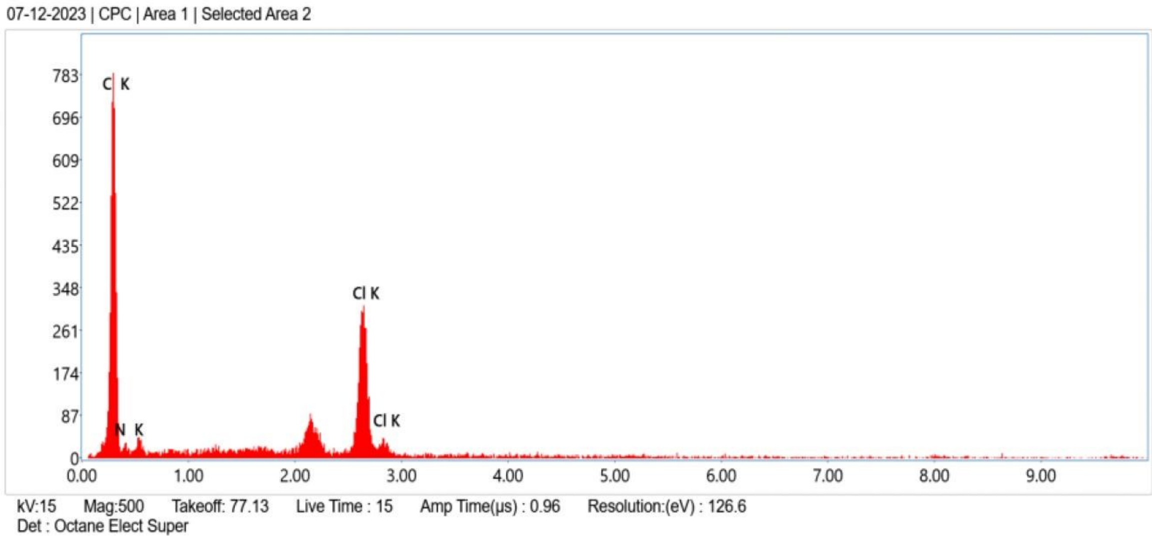

Figure 3: EDX spectra for carbon (C), nitrogen (N), and chlorine (Cl)

EDX is used to determine the relative amounts of these elements in the sample, providing an elemental composition. The weight percentage (wt%) and atomic percentage (at%) of each element will be provided. While hydrogen (H) cannot be detected by EDX, we can still compare the ratios of carbon, nitrogen, and chlorine to infer their presence in CPC. Elemental mapping is used to visually represent the distribution of chlorine and nitrogen in the sample. A uniform distribution of these elements would suggest a homogeneous dispersion of CPC, while an uneven distribution could indicate aggregation. The potential interference from coating materials (e.g., gold) used in sample preparation for FE-SEM was considered, as these elements can create peaks in the EDX spectrum and complicate the analysis of lighter elements like carbon and nitrogen. It is important to ensure that other compounds or materials do not interfere with the key elements of CPC (C, N, Cl). By weight, carbon (C) accounts for 87.4%, nitrogen (N) accounts for 5.4% and chlorine (Cl) for 7.2% by weight, as shown in Table 1.

Table 1: Analysis of elements carbon, nitrogen and chlorine in CPC with EDX

Elements	**Weight %**	**Atomic %**	**Error%**
CK	87.4	92.5	11.3
NK	5.4	4.9	33.4
Cl K	7.2	2.6	4.6

4.0 Conclusion

FE-SEM and EDX instruments were used to identify and quantify cetylpyridinium chloride, a popular and versatile surfactant. Detected CPC sizes ranged from 46.35 µm to 77.76 µm. The percentage of C, N, and Cl, in the sample of CPC under study, were found to be 87.4, 5.4, and 7.2 percent by weight respectively. There was less deviation of elemental composition from the theoretical value, showing the purity of the sample used.

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