Analysis of Percentage of Element of Mixed Surfactants in Mixed Solvent Media

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Abstract

Two surfactants interact with each other to form the mixed surfactant. Cationic surfactant cetyl pyridinium chloride (CPC) interacts with anionic surfactant sodium dodecyl sulphate (SDS) both at the air/water interface and in bulk, forming the ion-pair amphiphile (IPA) through the coulombic interaction between the binary surfactants with opposite charges. Energy-dispersive X-ray Spectroscopy (EDX) was used to identify and quantify the elements in the mixed surfactants and determine purity by calculating the percentage composition. The percentage composition of C, N, O, and S in the sample prepared in 10% methanol-water system was 32.0, 56.0, 13.5, and 2.5 respectively percent by weight.

Keywords: CPC, EDX, Mixed surfactant, SDS

Introduction

A surfactant (short for "surface-active agent") is a substance that reduces the surface tension between two liquids or between a liquid and a solid (Zhu et al., 2017). Surfactants' amphiphilic nature allows for easier mixing of non-mixing substances, such as oil and water (Yadav et al., 2024a). Surfactants are classified based on the charge of their hydrophilic (water-attracting) head group. The four main types of surfactants are anionic surfactants, cationic surfactants, nonionic surfactants, and amphoteric (Zwitterionic) surfactants (Kroll et al., 2022).

Anionic surfactants have a negatively charged head group. They are the most common type of surfactant and are known for their excellent cleaning and foaming properties as sodium lauryl sulphate (Ali et al., 2014). Cationic surfactants have a positively charged head group. They are typically used for their antimicrobial and conditioning properties, although they have weaker cleaning abilities compared to anionic surfactants such as cetyltrimethylammonium bromide, benzalkonium chloride, etc. Nonionic surfactants have no charge on their head group. They are less foaming than anionic surfactants but are very effective at emulsifying and are gentle, making them ideal for use in sensitive applications such as lauryl alcohol ethoxylate. Amphoteric (Zwitterionic) surfactants can carry both positive and negative charges, depending on the pH of the solution. They are mild and are often used in personal care products such as cocamidopropyl betaine (Lone et al., 2021).

Mixed surfactant refers to a combination of two or more different surfactant types that are blended to optimize their individual properties. When combined, the surfactants can work synergistically, resulting in improved performance compared to using a single surfactant. These blends are widely used in various applications to enhance characteristics such as cleaning power, foam formation, emulsification, or mildness (Bhattarai et al., 2013).

The combination of different surfactants can produce stronger effects than each surfactant alone. For instance, an anionic surfactant (which cleans effectively but may be harsh) can be combined with a nonionic or amphoteric surfactant (which is gentler) to balance performance and mildness. Mixed surfactants can have synergistic benefits, such as boosting surface activity, lowering surface tension, and increasing foamability (Bhattarai et al., 2017; Yadav et al., 2024b). Mixed surfactants can be categorized based on the types of individual surfactants that are combined to achieve synergistic effects. These combinations typically involve mixing surfactants from different classes—anionic, cationic, nonionic, and amphoteric—to optimize performance (Sachin et al., 2019).

In this study, we discuss and study the anionic + cationic type of mixed surfactants. Anionic + cationic surfactants are incompatible due to their opposite charges, which can lead to precipitation or reduced performance. However, under carefully controlled conditions, such mixtures can provide unique benefits such as improved emulsification or enhanced antimicrobial activity. These mixtures are less common in mainstream consumer products but can be found in specialized formulations like emulsions, disinfectants, or conditioning agents (Herrington et al., 1993). Such as sodium stearate (anionic) + quaternary ammonium compounds (cationic) which are used in certain emulsions and fabric softeners whereas alkylbenzene sulfonates + cetyltrimethylammonium bromide sometimes used in industrial or laboratory applications. In applications where excess foam can be problematic (e.g., industrial cleaning or wastewater treatment), a mixed surfactant system allows better control over foam production and stability (Bhattarai, 2015). Mixed surfactants are more effective at stabilizing emulsions, such as oil-in-water or water-in-oil systems, which are common in cosmetics, food products, and pharmaceuticals.

Sodium dodecyl sulphate is an anionic surfactant (Niraula et al., 2022). There are many applications for sodium dodecyl sulphate (SDS), including cleaning products, hygiene products, and biochemical research. Cetylpyridinuim chloride (CPC) is a cationic surfactant. Combining CPC surfactant with chlorhexidine, can overcome its clinical limitations or drawbacks without changing or reducing the antimicrobial activity (Al-Sada & Al-Gharrawi, 2024).

SDS and CPC can be used to create coacervates, which are colloidal systems formed by the electrostatic interaction of oppositely charged surfactants. These coacervates can encapsulate active ingredients for delivery in various industries such as food, pharmaceuticals, and cosmetics. It has been shown that cetyl pyridinium chloride (CPC) interacts with sodium dodecyl sulphate (SDS) both at the air/water interface and in bulk, forming the 'catanionic coacervate' or ion-pair amphiphile (IPA) through the coulombic interaction between the binary surfactants with opposite charges. A ternary phase behavior study of the system CPC/SDS/H_2O was conducted (Maiti et al., 2010). SDS is anionic (negatively charged) due to its sulphate group. CPC is cationic (positively charged) because of its quaternary ammonium group. When mixed, the negatively charged SDS molecules can interact with the positively charged CPC molecules, leading to ionic neutralization. This can result in precipitation (Kume et al., 2024).

Energy-dispersive X-ray Spectroscopy (EDX) is a powerful analytical technique used to determine the elemental composition of a sample. When applied to a mixed surfactant system like sodium dodecyl sulphate (SDS) and cetylpyridinium chloride (CPC), EDX provides insights into the chemical elements present and how they are distributed within the sample (Scimeca et al., 2018). EDX displays the X-rays collected during any one analysis period at mid-energy (1-20 keV) simultaneously (Morgan, 1985). An X-ray spectrum shows the energy of the rays as a histogram plot. Both qualitative and quantitative data are contained in the spectrum of EDX microanalysis. An element is identified by the position and energy of a peak in the spectrum; the area under the peak is proportional to the number of atoms of the element. An electron beam can also produce X-rays when it is slowed by the electrostatic fields of atomic nuclei. There is a continuous radiation of X-rays below the peaks of this spectrum. Analyzing a spectrum qualitatively, i.e. identifying elements, is usually accomplished with software provided by the manufacturer. The EDX technique is conventionally used for elemental analysis of samples' surfaces. In elemental analysis, this method has some limitations. X-ray spectrometry only identifies elements and cannot discriminate between ionic and nonionic species. Furthermore, the EDX requires that all samples be examined under nearly vacuum conditions, which has major consequences for specimen preparation because electrons and X-rays are strongly absorbed by air molecules. In general, X-ray detection is not affected by the chemical state of elements, but rather by inter-element interference, known in X-ray spectrometry as peak overlap, which causes major problems in elemental analysis. As a result, it is possible to identify atoms with an atomic number greater than 10. The minimum detectable elemental concentration, which necessitates some signal averaging, is around 0.1 mmol per kg of dry specimen (10 ppm), with spatial resolution ranging from around 10 nm to a few micrometers (Pivovarova et al., 2013). To reduce the detection limit, more counts are required, which can be obtained by increasing the counting time and/or beam current.

The EDX microanalysis enabled elemental analysis on several isotypes of calcification and provided further information on the relationship between calcification and disease (Scimeca et al., 2018). Furthermore, EDX enabled tremendous progress in understanding the molecular processes and effects of calcification. For example, Sonou et al. reported for the first time on the reliable assessment of the chemical composition of aortic medial calcification using the SEM-EDX (Sonou et al., 2015). A study examined the role of CPC in inhibiting mild steel corrosion in acidic media. As part of the study, weight loss and potentiodynamic polarization methods were employed to measure inhibition efficiency, as well as FTIR, FESEM, EDX, and AFM, to observe changes on the surface (Yadav et al., 2024c). With the help of the EDX spectrum of a nanostructured surfactant such as sodium dodecyl sulphate (SDS) and cetyltrimethylammonium bromide (CTAB) activated mixed metal oxide nanomaterial (zinc oxide/nickel oxide), the elemental composition was revealed (Chellamuthu et al., 2024). There was no study of EDX of mixed surfactant (CPC-SDS).

It is our objective to observe the analysis of EDX in 10% methanol-water mixed solvent media with a mixture of 0.0l M sodium dodecyl sulphate (SDS) and 0.01M cetyl pyridinium chloride.

Materials and Methods

The materials used in this study were sodium dodecyl sulphate (SDS) and cetylpyridinium chloride (CPC). Sodium dodecyl sulphate (SDS) and cetylpyridinium chloride (CPC) were purchased from HIMEDIA, India. EDX (Model no: Elect Super Company, USA) was used for the characterization technique.

The solutions of 0.0l M SDS and 0.01 M CPC were prepared separately in 250 ml volumetric flasks using 10% methanol-water mixed solvent media. The accurate weight of 0.072 gm of SDS and 0.895 gm of CPC was weighed using a four-digit weight balance. Then these two solutions were mixed by adding equal volumes of each other. The precipitate was obtained from cetyl pyridinium chloride and sodium dodecyl sulphate. The precipitated material was further separated by filtration. The remaining residue was washed repeatedly until NaCl was removed from the sediment and tested with AgNO3 solution; the residue was dried for 24 h at 60 °C temperature in the oven. White powder was obtained from the mixture of CPC and SDS when the weight was reduced.

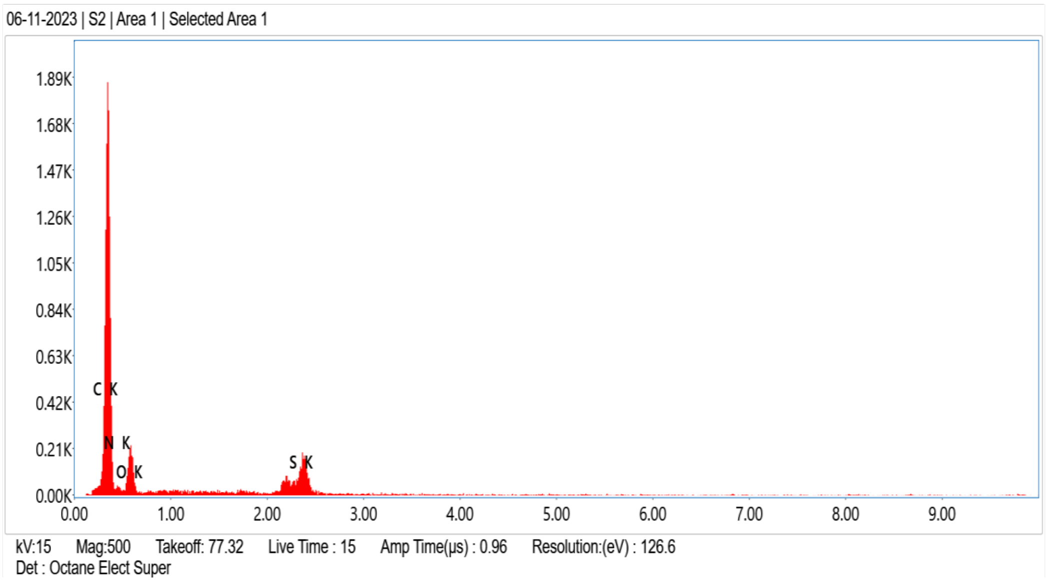
Results and Discussion

To analyze the percentage of the element of SDS-CPC in 10% methanol-water mixed solvent media, 0.01 M sodium dodecyl sulphate (SDS) and 0.01M cetylpridinium chloride were mixed with concentrations of SDS and CPC above the critical micelle concentrations of 0.0082 M SDS (Niraula et al., 2018) and 0.0012 M CPC (Shahi et al., 2024) have taken in our research work. The reason for taking 0.01M concentration for both SDS and CPC surfactants is to make precipitation in 10% methanol.

EDX analysis is applied to the SDS + CPC mixed surfactant system in 10% methanol as shown in Figure 1. The **EDX spectrum** shows peaks corresponding to different elements in the SDS + CPC system. The key elements that EDX detect include Sulfur (S) from the sulphate group in SDS, Sodium (Na) from the sodium cation in SDS, Chlorine (Cl) from the chloride ion in CPC, Nitrogen (N) from the quaternary ammonium group in CPC, Carbon (C) and Oxygen (O) from the alkyl chains and functional groups in both surfactants (Jia et al., 2019). EDX can provide a quantitative analysis of the relative abundance of these elements in the SDS-CPC mixture. This is important for confirming the **stoichiometry** of the mixed surfactant system. For example, we can determine if the expected ratios of sodium to sulfur (from SDS) or nitrogen to chlorine (from CPC) are correct. It is a feature of EDX that shows the distribution of specific elements across the sample. This is useful in analyzing how SDS and CPC interact at the molecular level. If the surfactants are forming separate phases or precipitating due to charge neutralization, the elemental maps may show regions of higher concentrations of sulfur and sodium (indicating SDS-rich areas) and regions with higher concentrations of nitrogen and chlorine (indicating CPC-rich areas). When SDS and CPC interact (due to their opposite charges), they can form ionic complexes. EDX can help confirm the formation of these complexes by showing the co-localization of elements such as sulfur (from SDS) and nitrogen or chlorine (from CPC). Precipitation of these ionic complexes may also be visible in the elemental maps, where the mixed surfactants form solid particles with a distinct elemental composition.

Figure 1

EDX analysis of CPC+SDS in 10% methanol



In Figure 1, the X-axis is the number of elements present in the sample and the Y-axis is the percentage of that element which helps to compare for other elements. EDAX APEX is a software program used for collecting and analyzing EDX data.

Comparing EDX spectra of pure SDS, pure CPC, and the SDS-CPC mixture can show how their elemental composition changes upon mixing. For example, the formation of precipitates may result in a decrease in the signal of free sodium ions (from SDS) or free chloride ions (from CPC), indicating that these ions are now part of a complex structure. The EDX analysis confirms the elements present in both surfactants (e.g., sulfur, sodium, chlorine, nitrogen, carbon, and oxygen). This helps verify the identity of the surfactants and ensures that the correct components are present in the mixture (Loginova et al., 2019). EDX helps confirm the relative ratios of the elements present in SDS and CPC. This is particularly useful for determining whether the mixing of SDS and CPC leads to precipitation or complex formation, as evidenced by changes in the expected ratios (e.g., sodium to sulfur or nitrogen to chlorine). If precipitation occurs due to charge neutralization between SDS and CPC, EDX analysis will show the formation of distinct elemental regions corresponding to the precipitated complexes. These regions will have a co-localization of sulfur (from SDS) and nitrogen or chlorine (from CPC), indicating that the surfactants have formed ionic complexes. EDX can reveal if phase separation occurs, where SDS-rich and CPC-rich areas are observed separately. For instance, sulfur and sodium may dominate one area (SDS phase), while nitrogen and chlorine dominate another (CPC phase), which can indicate limited miscibility between the two surfactants.

If the SDS and CPC mixture is homogeneous, the elemental distribution maps from EDX will show a uniform distribution of the key elements (S, Na, N, Cl) across the sample. On the other hand, inhomogeneous mixtures will show clustering of certain elements, indicating incomplete mixing or localized complex formation.

EDX can confirm the presence and distribution of both SDS and CPC in a formulated product. This is useful in industries like pharmaceuticals, cosmetics, and cleaning products, where precise control over the surfactant composition is crucial. EDX helps identify the formation of ionic complexes or precipitates due to surfactant interactions. Understanding these interactions is key to controlling product stability and performance, such as in drug delivery systems or emulsions. By analyzing the elemental ratios and distributions, EDX can provide feedback on how to optimize the mixing ratio of SDS and CPC to achieve the desired performance, such as maximum surfactant efficiency or optimal antimicrobial action.

Table 1

Elemental composition data of CPC+SDS in 10% methanol-water system

Element	Weight %	Atomic %	Error %
C K	32.0	34.2	10.8
N K	56.0	54.8	11.4
O K	13.5	12.9	4.5
S K	2.5	1.1	4.9

Table 1 displays the elemental composition data for CPC+SDS in 10% methanol-water mixed solvent media. Carbon (C) appeared at around 32.0 % by weight. Nitrogen (N) appeared at around 56.0%, Oxygen (O) appeared at around 13.5% and Sulphur (S) appeared at around 2.5% by weight. However, the atomic percentage of carbon is 34.2%, nitrogen is 54.8%, oxygen is 12.9%, and sulfur is 1.1% in the analyzed sample of CPC+SDS mixture which was prepared in 10% methanol. The percentage of error provided by the EDX instrument. It suggests that it is a variation of composition in the sample when the test repeats again and again in the same instrument and same sample.

Conclusions

EDX was used for the identification and quantification of the elements present in the mixed surfactant CPC+SDS to determine the purity of CPC+SDS by calculating the percentage composition. The percentage composition of C, N, O, and S in the sample prepared in 10% methanol-water system is found to be 32.0, 56.0, 13.5, and 2.5 respectively percent by weight. The results revealed that all elements (C, N, O, S) are present in CPC+SDS. This information is valuable for applications in various industries, including pharmaceuticals, personal care, and materials science. Indeed, EDX analysis could be a powerful technique for investigating heavy metal buildup in tissues and in forensic science as well as investigating the harmful effects and potential drug delivery of NPs.

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