Chapter IV

SCANNING ELECTRON MICROSCOPE WITH ENERGY DISPERSIVE X=RAY SPECTROMETER (SEM WITH EDS)

Introduction

The study of surface morphology by scanning electron microscopy reveals the presence of metallo dyes on the surface of the membranes, the size of the particles and the magnification power being very high. The scanning electron microscope (SEM) is equipped with energy dispersive X-ray spectroscopy (EDS) and it has been extensively used. SEM has the advantage of providing high-resolution images that describe the micro morphology of specimens. It is often necessary to identify the different elements associated with a specimen[1, 2]. This is accomplished by using the 'built-in' spectrometer called Energy Dispersive X-ray Spectrometer. EDAX is an analytical technique with utilizes X-ray that are emitted from the specimen when bombarded by the electron beam to identify the elemental composition of the specimen. When the electron beam of the SEM bombards the sample, electrons are ejected from the atoms on the specimen's surface[3-6].

Emission spectral analysis methods were used invariably in all branches of Industry, Ecology, Geology, Agriculture, Medicine, and Biology etc. SEM-EDAX is a well-established analytical tool for simultaneous multi-elemental determination of minor, major and trace elements in various samples. SEM-Energy dispersive spectrometry has been extensively used in the analysis of minor, major and trace elements in biological materials because identification of chemical constituents in different regions of the sample without destroying it and also when the sample is small. Same portion of the sample can be used for the other type of studies like Infrared Spectroscopy, X-ray diffraction. A resulting electron vacancy is filled by an

electron from a higher shall, and an X-ray is emitted to balance the energy difference between the two electrons The EDA X-ray detector measures the number of emitted X-rays versus their energy. The energy of the X-ray is of the energy versus relative counts of the detected-ray is obtained and evaluated for qualitative and quantitative determinations of the elements present in the samples[7, 8].

Discussion

The morphology of the metallo dyes showed some important observations. Typical SEM photographs are shown in Fig. 4.1-4.4. Coverage of surface of the adsorbent due to adsorption of the adsorbate molecules presumably leading to formation of a monolayer of the adsorbate molecule over the adsorbent surface is evident from the formation of white layer (molecular cloud) of uniform thickness and coverage (spread).The above observation was further confirmed well with the batch mode adsorption studies. SEM studies visualized the formation of the molecular cloud of the metallo dyes over surface.

SEM studies (Fig 4.1- 4.4) reveal the change in the morphology of particles and homogeneity of the product. Even if there is a clear difference in the crystallization state of the metallo dyes samples (not for all), viz., Amaranth, Methylene blue, Remazol red B and Golden yellow HER of lead, cobalt, nickel and copper complexes , the corresponding EDAX are also given. As an illustration of many of these SEM images obtained with magnification of x 5000 (fig. 4) shows a big size elliptic particle[9-12] .

EDS Analysis

Semi quantitative estimation of major and minor elements present in the samples has been done under SEM, along with EDAX. The energy value of each peak may be matched with X-ray emission wavelength for non-diffractive analysis

and the elements present in metallo dyes (except Lead and Cobalt complexes due to their low percentage).

Summary and Conclusion

In the present investigation, the SEM/EDAX studies have been carried out in the flowing systems.

A scanning electron microscope is used to take the surface photographs and surface characteristics are reported. Using JSM-5610LV operated at 20 KV.

Semi quantitative estimation of major and minor elements present in the samples has been done under SEM, along with EDS. The energy value of each peak is matched with X-ray emission wavelength for non- diffractive analysis and the elements present in the metallo dyes are identified.

References

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