



X-RAY, SEM, EDAX AND THERMAL ANALYSIS OF WATER DISSOLVED SOLIDS

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ABSTRACT

Water dissolved solids have been extracted by conventional boiling and filtering technique. The extracted solids have been characterized by X-ray diffraction, Scanning electron microscopy (SEM), energy dispersive X-ray microanalysis (EDAX), thermogravimetric analysis (TGA) and dc electrical conductivity measurement methods. Comparison of the results of the as extracted and heat treated (up to 1200°C) samples show that the heat treated composite sample is thermally very stable, showing very high value of electrical resistivity almost temperature independent and hence, showing very high dielectric strength, useful for high temperature dielectric devices.

Key words : X-ray diffraction, SEM, EDAX, TGA, dc conductivity

INTRODUCTION

Udaipur, the lake city of Rajasthan surrounded by Aravali hills is facing water crisis due to minimum rainfall for the last 5 years. Due to increasing industrialization, urbanisation and other developmental activities as well as unplanned and excessive exploitation and mounting anthropogenic influences in and around aquatic ecosystem have resulted in pollution problems. Lakes being fragile ecosystem are vulnerable to such problems. Pollution caused by human activities affects the physico-chemical characteristics of water¹⁻³, leading to destruction of community health and deteriorating the lake environment. After careful analysis of the domestic supply of water, it is found that the total dissolved solids (TDS) have crossed the standard permissible limit⁴ responsible for different water borne diseases/disorders⁵.

The aim of the present paper is to extract the total dissolved solids from the domestic supply water and characterise them using X-ray diffraction, scanning electron microscopy (SEM), energy dispersive X-ray microanalysis (EDAX), thermogravimetric analysis (TGA) and dc electrical conductivity measurements.

EXPERIMENTAL

Extraction of dissolved solids

Water samples collected from the domestic supply were boiled for half an hour in a steel container of 5 litres capacity and kept for settling for 24 hours and then filtered through the usual filtering technique. The dissolved solids were filtered, dried and obtained into very fine powder form of white colour.

The powder sample was pressed into pellets of 1.2 cm diameter by applying 5 tonnes of pressure in a die punch system using a hydraulic press. After pelletization, the pellet samples were sintered at 1200°C in air and cooled to room temperature. The sintered pellet was electroded using silver paint for measuring its conductivity.

Measurements

The X-ray diffractograms of the as extracted raw powder and calcined (heat treated) at 1000°C and 1200°C were recorded using Philips X-ray diffractometer at a scanning rate of 2°/minute in a wide range of 2 θ from 20° to 100° using Cu k_{α} radiation and nickel filter.

Scanning electron micrograph and EDAX measurements were done on a Philips XL-30 ESEM Scanning electron microscope linked with EDAX system. The micrographs were taken at different spots having magnifications of 2000X, 3000X and 4000X applying 20 KV accelerating voltage. The EDAX analysis was done to check the chemical composition of the material applying the accelerating voltage of 22 KV. Thermogravimetric analysis was carried out from room temperature (RT) to 1000°C in air using laboratory made set up comprising of high precision electronic balance, model MODERN (M/1 PM) and high precision METREX furnace which can go up to temperature of 1400°C. DC electrical conductivity was measured from RT to 650°C on the pellet sintered at 1200°C by two probe method. The dc current was measured using a high precision digital nanoammeter (Model : NAM-100) applying 6V dc across the sample. The temperature was measured using a digital temperature indicator calibrated with Chromel-Alumel thermocouple.

RESULTS AND DISCUSSION

X-Ray diffraction

The X-ray diffractograms of the as extracted raw powder as well as heat treated at 1000°C and 1200°C powder are shown in the Figure 1 [a,b,c]. In Fig. 1[a] a large number of peaks, comprising salts of Ca, Mg, Al, Si, Mo, P etc. mostly in the form of oxides, hydroxides, carbonates, chlorides, silicates, phosphates and their combinations appeared. When the same powder was heated at 1000°C, it decomposes into its more stable powder form and hence a large

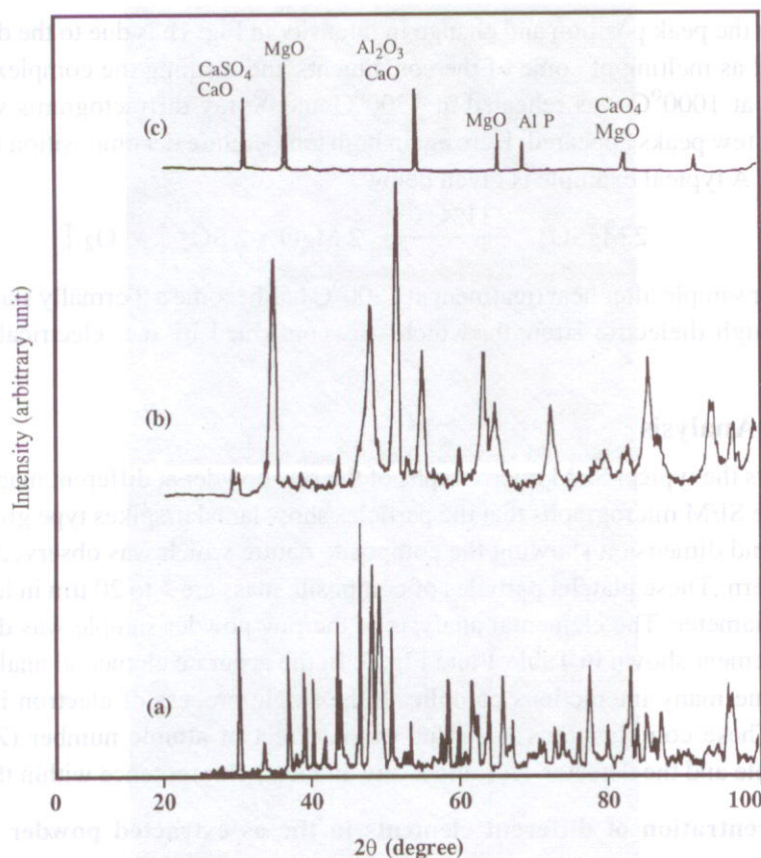
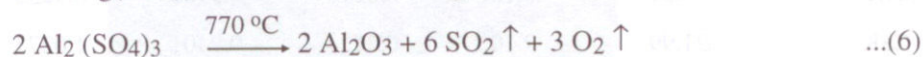
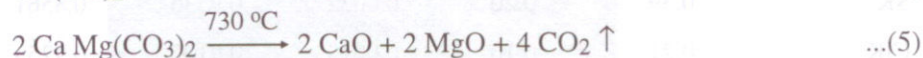
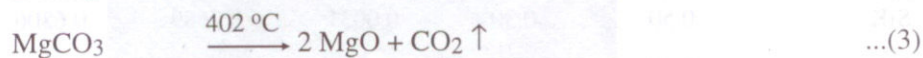
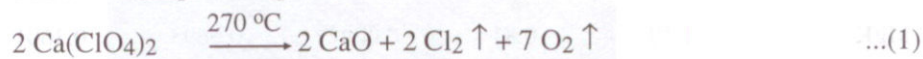


Fig.1. X-ray diffraction patterns of (a) as extracted raw powder and heat treated powder at (b) 1000°C and (c) 1200°C

number of peaks disappeared and some are shifted. The following probable decomposition reactions confirmed the decomposition process.



The shift in the peak position and change in intensity in Fig. 1b is due to the decomposition reaction as well as melting of some of the constituents and forming the complexes. When the powder heated at 1000°C was reheated at 1200°C and X-ray diffractograms were recorded [Fig. 1(c)] only few peaks appeared. Here again high temperature decomposition reaction/melting takes place. A typical example is given below:



The powder sample after heat treatment at 1200°C has become a thermally stable composite mass of very high dielectric strength, which was confirmed by d.c. electrical conductivity measurement.

SEM, EDAX Analysis

Fig. 2 shows the typical SEM micrographs of the raw powder at different magnifications. It is clear from the SEM micrographs that the particles show lamellar/spikes type growth that vary in shape, size and dimension showing the composite nature which was observed in the X-ray diffraction pattern. These platelet particles of composite mass are 5 to 20 µm in length and less than 2 µm in diameter. The elemental analysis of the raw powder sample was done using the EDAX measurement shown in Table 1 and Fig. 3. In the accurate elemental analysis using the EDAX technique many interactions complicate the single process of electron induced x-ray fluorescence. These complications arise due to the effect of atomic number (Z), absorption within the sample and the detector (A) and x-ray induced fluorescence within the sample (F).

Table 1. Concentration of different elements in the as extracted powder from EDAX analysis

Element	Weight %	Atomic %	K-Ratio	Z	A	F
CK	32.99	45.57	0.1312	1.0254	0.3875	1.0005
OK	41.84	43.39	0.0572	1.0089	0.1356	1.0001
MgK	1.19	0.81	0.0042	0.9693	0.3601	1.0012
AlK	0.68	0.42	0.0031	0.9411	0.4908	1.0022
SiK	0.50	0.30	0.0031	0.9689	0.6306	1.0039
PK	0.20	0.11	0.0014	0.9371	0.7550	1.0072
SK	0.39	0.20	0.0032	0.9536	0.8561	1.0126
ClK	0.21	0.10	0.0018	0.9100	0.9253	1.0219
CaK	21.99	9.10	0.2124	0.9401	1.0273	1.0000
Total	100.00	100.00				

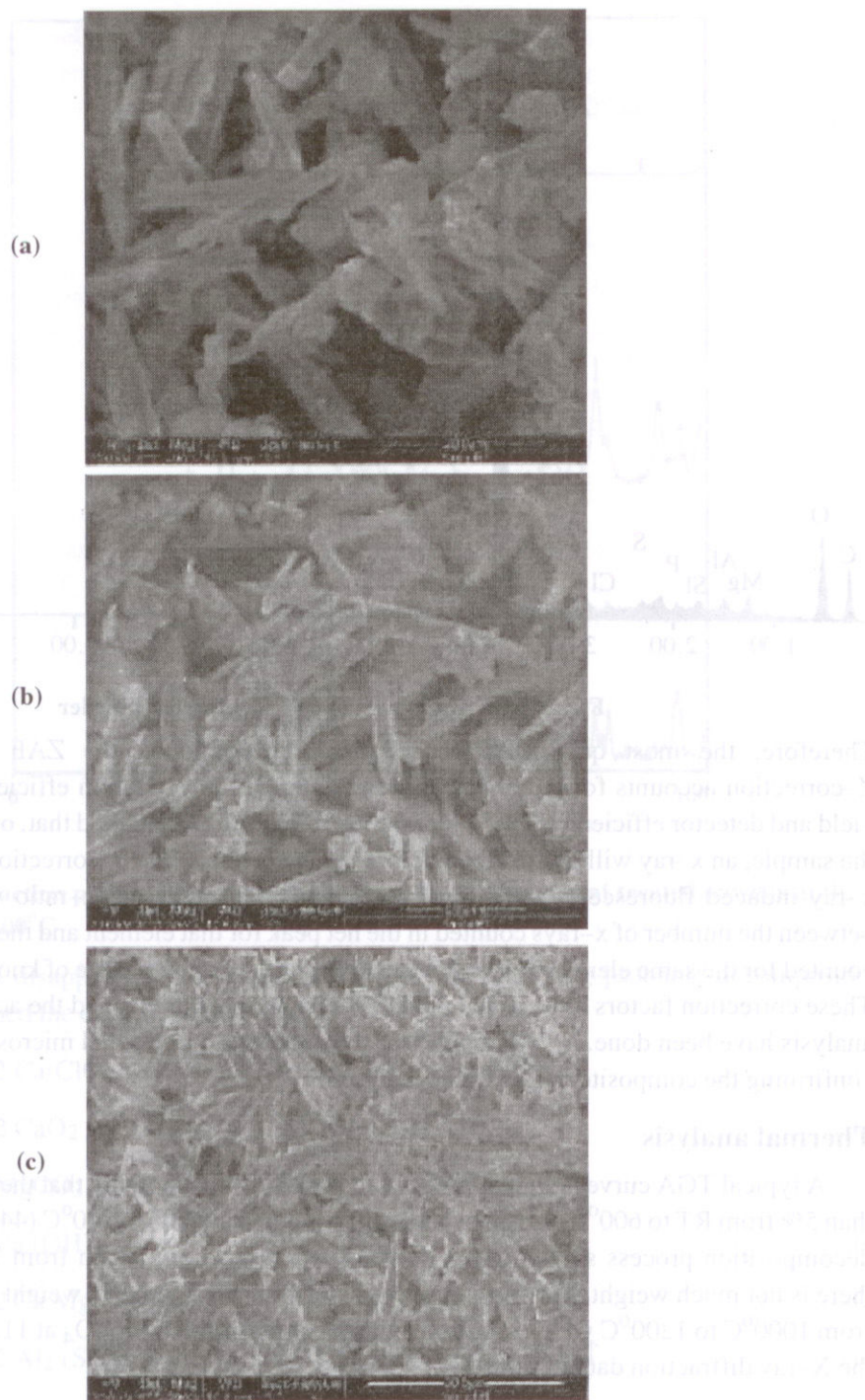


Fig. 2. SEM micrographs of as extracted powder (a) 4000 X (b) 3000 X and (c) 2000 X

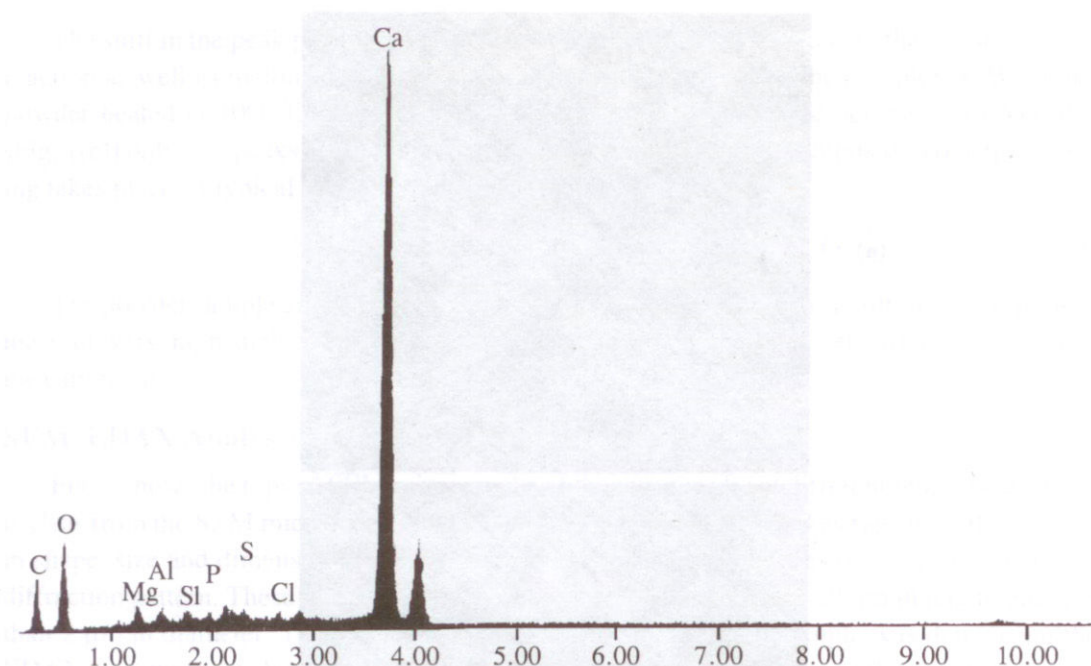


Fig. 3. EDAX diagram of the as extracted powder

Therefore, the most quantitative calculation depends upon the ZAF corrections. The Z-correction accounts for the effect of atomic number on excitation efficiency, fluorescence yield and detector efficiency. The A-correction reflects the likelihood that, once created within the sample, an x-ray will be absorbed before being detected. The F-correction takes care of the x-ray induced fluorescence. The ZAF corrections are applied to k-ratio which is the ratio between the number of x-rays counted in the net peak for that element and the number of x-rays counted for the same element under the identical condition in a sample of known concentration. These correction factors have been applied, shown in the Table 1, and the accurate elemental analysis have been done. EDAX analysis also supports the x-ray and microstructural analysis, confirming the composite nature of the material.

Thermal analysis

A typical TGA curve is shown in Fig. 4. It is clear from the figure that the weight loss is less than 5% from RT to 600°C, which becomes maximum from 600 to 700°C (44%) confirming the decomposition process shown in the decomposition reaction. Again from 700°C to 1000°C, there is not much weight loss. Furthermore, an appreciable change in weight loss was observed from 1000°C to 1200°C which confirmed the decomposition of MgSO_4 at 1124°C and supports the X-ray diffraction data of Fig. 1(c).

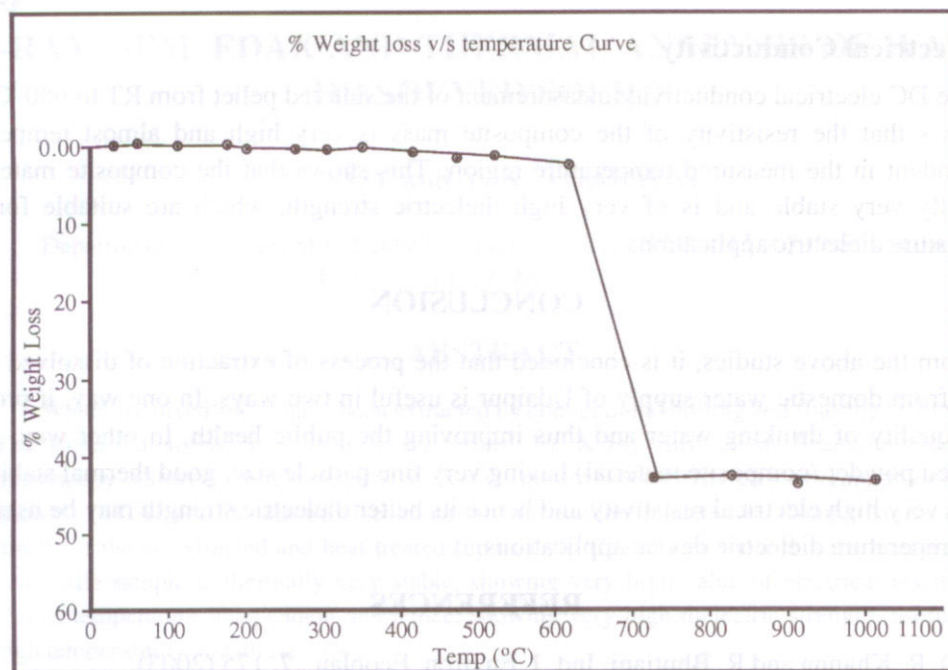
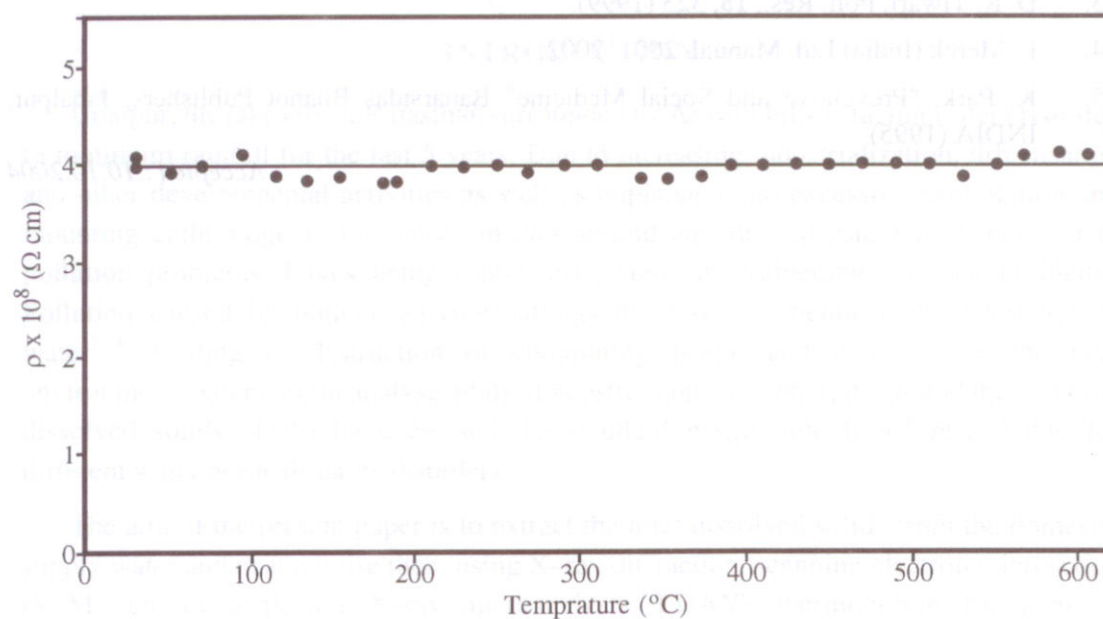


Fig. 4. TGA Curve

Fig. 5. ρ v/s T curve of the sintered pellet at 1200°C

DC Electrical Conductivity

The DC electrical conductivity measurement of the sintered pellet from RT to 650°C (Fig. 5) shows that the resistivity of the composite mass is very high and almost temperature independent in the measured temperature region. This shows that the composite material is thermally very stable and is of very high dielectric strength, which are suitable for high temperature dielectric applications.

CONCLUSION

From the above studies, it is concluded that the process of extraction of dissolved solids (salts) from domestic water supply of Udaipur is useful in two ways. In one way, it provides better quality of drinking water and thus improving the public health. In other way, as the extracted powder (composite material) having very fine particle size, good thermal stability as well as very high electrical resistivity and hence its better dielectric strength may be useful for high temperature dielectric device applications.

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