

## PREPARATION & DIELECTRIC STUDY OF UNDOPED SODIUM SILICATE

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Sodium Silicate ceramic was prepared by solid-state reaction technique. The formation of the compound was confirmed using X-ray diffraction technique at room temperature. The dielectric constant ( $\epsilon_r$ ) and the dielectric loss ( $\tan\delta$ ) have been measured at different temperatures for frequencies up to 5 MHz. Detailed studies of dielectric constant ( $\epsilon_r$ ) and loss tangent ( $\tan\delta$ ) were done as a function of frequency (100 kHz-5 MHz) at 70 °C temperatures and up to temperature 450 °C by using a Hioki 3532 LCR- Hi-tester. The analyses for this sample indicates that dielectric constant variation with frequency exhibits an initial drop in the lower frequency region followed by a nearly frequency independent behaviour in the high frequency region, which confirms the normal behaviour of dielectric materials. Detailed studies of dielectric properties of the compound as a function of temperature (70-450 °C) at different frequencies (100 kHz -5 MHz) suggest that the compound has multiple dielectric anomalies in the studied temperature and frequency ranges.

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### 1. Introduction

Sodium Silicate ( $\text{Na}_2\text{SiO}_3$ ) is an ideal system to study the dielectric behaviour. This work is aimed to prepare the sample via solid-state reaction technique and to study the dielectric properties of the prepared sample.

The dielectric studies were done by varying frequency with temperature as parameter. Both the real and the imaginary parts of the dielectric constant depend on frequency as well as temperature. Both components show strong dispersion at low frequencies which is associated with good ionic conductivity, because at high frequencies the charge carriers fail to respond to the external field, therefore the measured dielectric constant is mainly from the intrinsic polarization. The dielectric constant also depends on temperature. The dielectric loss in the low frequency region is very high compared to that in the high frequency region. But the dielectric loss increases with increasing temperature [1-8].

### 2. Experimental

Undoped Sodium Silicate was prepared using a high temperature solid-state reaction technique using high purity (99.9%) ingredients  $\text{Na}_2\text{CO}_3$  and  $\text{SiO}_2$  in a suitable stoichiometry. For Preparation of 100gm  $\text{Na}_2\text{SiO}_3$  we have taken the constituent compounds in the following weight %

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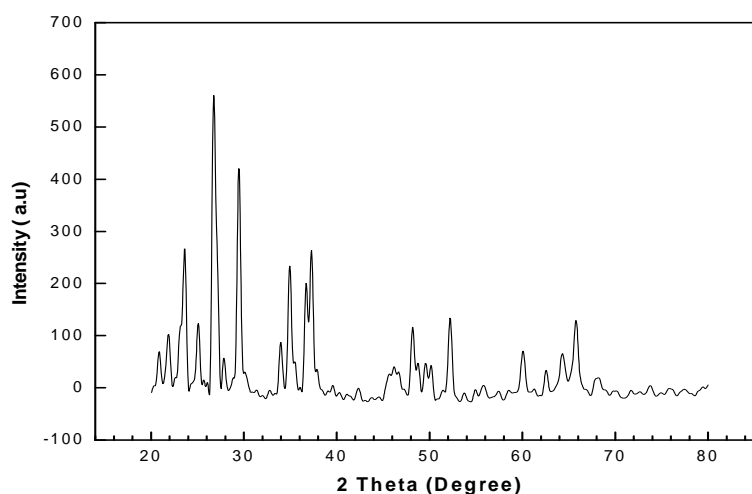
Sample	Na <sub>2</sub> CO <sub>3</sub>	SiO <sub>2</sub>
Undoped	58.19	65.963

The powder sample was dried so that there is no possibility of moisture absorbed on the surface of the grains of ingredients, which cause significant errors in measuring the correct weight for samples. The constituent compounds, in suitable stoichiometry, were thoroughly mixed in an agate mortar for 2h. Mixing was done using acetone as mixing medium. The mixed materials were then calcined in a quartz crucible at 900 °C for 2 h. The process of grinding and calcinations was repeated till homogeneous powder of the desired compound was obtained. The formation and quality of the compound were checked by X-ray diffraction (XRD) technique with CuK<sub>α</sub> radiation. The fine and homogeneous powder of the compound was used to make cylindrical pellets of diameter 30 ~ mm and thickness 5 ~ mm by applying pressure using a hydraulic press. Polyvinyl alcohol (PVA) was used as a binder to prepare pellets. The organic binder PVA was burnt out during the sintering at 700 °C for 2 hr in an air atmosphere. The sintered pellets were polished and then electrode by high-purity ultra-fine silver paste for electrical measurements. The relative dielectric constant ( $\epsilon_r$ ) and loss tangent ( $\tan\delta$ ) of samples were obtained as a function of frequency at different temperatures using a HIOKI 3532 LCR Hi-tester meter (Japan) with a laboratory-made sample holder. The permittivity ( $\epsilon'$ ) and loss tangent ( $\tan\delta$ ) of them were calculated, Plotted and commented on.

### 3. Results and discussion

#### 3.1 Structural analysis

The XRD pattern of the sintered Sodium silicate sample is shown in Fig.1. For powder X-ray diffraction a 2 – circle Goniometer, working in Bragg-Brentano geometry, was used with a rotating anode X-ray generator. Fig.1 shows the XRD pattern of Sodium silicate sintered at 700 °C. Here we can see that the diffraction peak is little sharper and symmetric. This shows that complete calcinations have occurred [2,6 and 7].

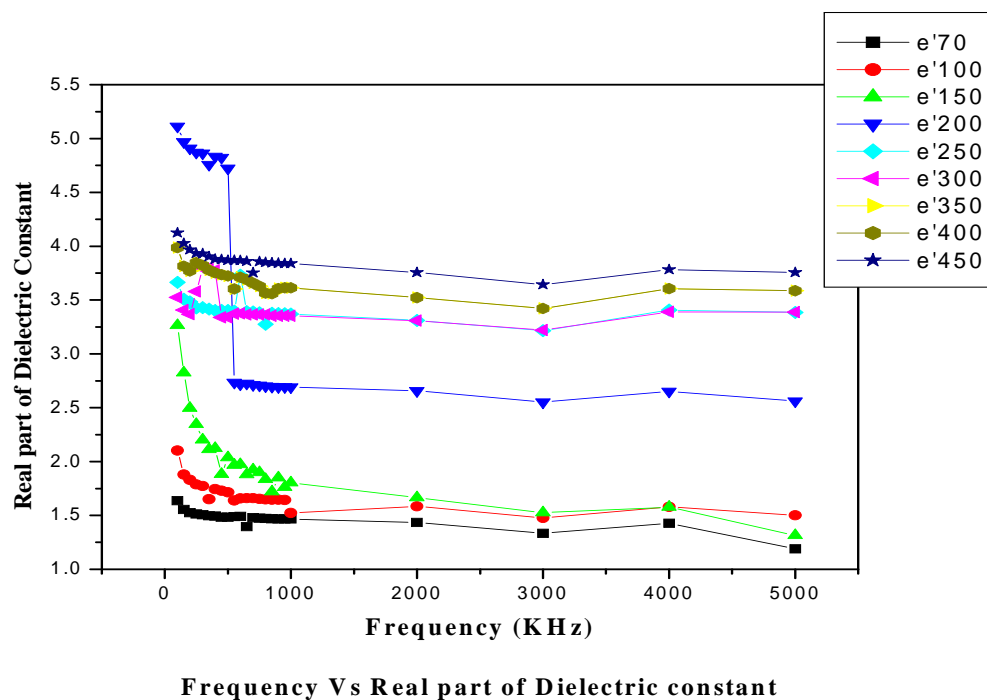


*Fig.1 Room temperature XRD pattern of undoped Sodium silicate.*

### 3.2 Dielectric Studies

The change in dielectric constant ( $\epsilon$ ) and dielectric loss ( $\tan\delta$ ) of Sodium Silicate with frequency (100 KHz-5MHz) at different temperatures are shown in Fig.2, 3 & 4. The dielectric constant decreases with increasing frequency. At lower frequency the dielectric dispersion is large and it becomes independent of frequency above 1 MHz This is due to the fact that the dielectric material exhibits induced electric moment under the influence of electric field. At higher frequency, the polarization of the induced moments could not synchronize with the frequency of applied electric field. So, the dielectric attains a constant value above certain frequencies. At low frequencies, normally all types of polarizations exist. However, in Sodium Silicate, the ionic and electronic polarizations exist in the high-frequency range. It is observed that at higher frequencies these parameters become almost frequency-independent [1, 2, and 5].

The dielectric Constant has higher values at low frequencies and also at high temperature. For undoped sodium silicate the dielectric constant also depends on temperature. With increase in temperature the dielectric constant increases. Because at higher temperatures the coupling between the charge carriers are very prominent and due to this reason the dielectric constant increases with temperature. At about 300° C there is an anomaly observed in the dielectric constant .This may be due to phase transition at this temperature [1, 2, 6, and 7].



*Fig.2 Variation of Real part of Dielectric constant ( $\epsilon'$ ) of Sodium silicate as a function of frequency*

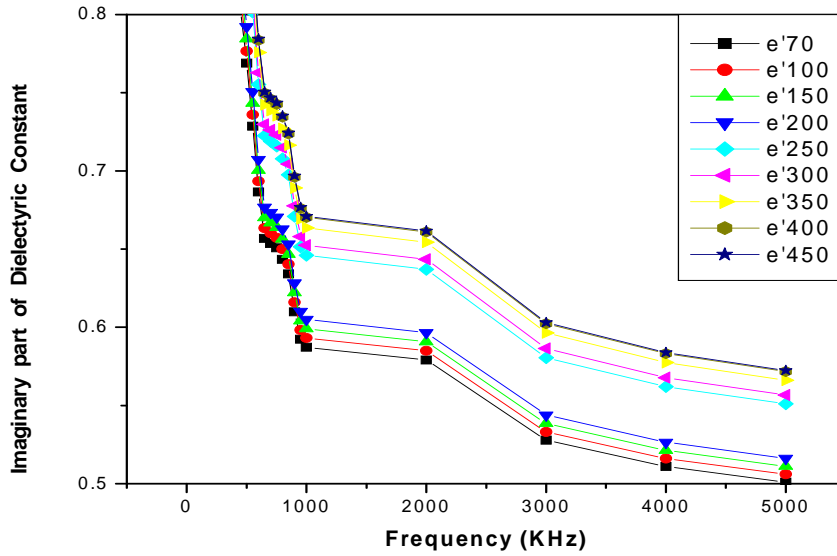


Fig.3 Variation of imaginary part of Dielectric constant ( $\epsilon''$ ) of Sodium silicate as a function of frequency

### 3.3 Dielectric Loss

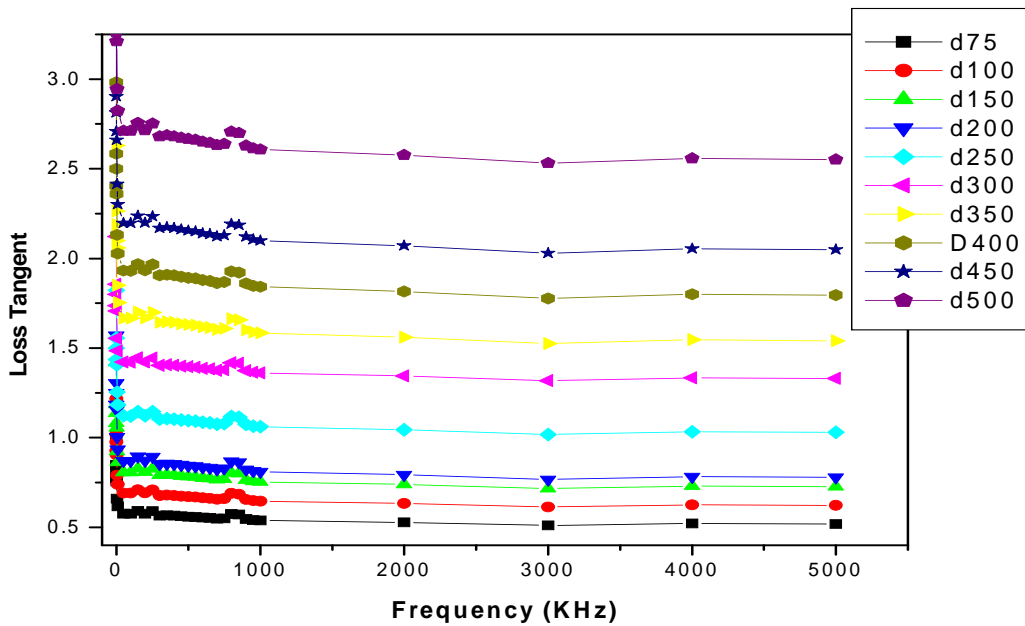


Fig.4. Variation of dielectric loss ( $\tan\delta$ ) of Sodium silicate as a function of frequency at different temperatures.

The dielectric loss in the low frequency region for all temperatures are very high and decreases with increasing frequency indicating a normal behaviour of dielectric materials having mobile charge carriers of both types of both types (i.e., ions and electrons). The trend in  $\tan\delta$  versus frequency curve might be attributed to parallel conduction ( $\tan\delta = 1/\omega CR$ ), where  $\omega$  is the

angular frequency, C the capacitance and R is the resistance), which is probably due to porosity. The loss tangent is a useful indicator of the potential of a material to extract energy from an electromagnetic field. The increase in permittivity and loss tangent, observed at room temperature as a function of frequency, decreases and can be ascribed to the mobility enhancement of the ionic charge carriers. But as we go on increasing the temperature the dielectric loss will also increase [1, 2, 5, 6, and 7].

#### 4. Conclusions

The dielectric constant shows strong dispersion at low frequencies. This dispersion in the imaginary part of the dielectric constant is stronger than that in the real part. The dielectric Constant also depends on temperature. It shows a strong dielectric dispersion starting from 300°C and increases with increase in temperature. The dispersion is stronger near 1 KHz. The anomaly in the increase of dielectric Constant ( $\epsilon$ ) at one kHz frequency under study at all temperatures is a consequence of phase transition. The dielectric loss in the low frequency region is very high than that in the high frequency region. In the high frequency region the permanent dipoles fails to respond readily to the electric field variations and hence the contribution to the Dielectric Loss decreases to a constant value. The sharp fall in the dielectric loss near 100 KHz is due to phase transition. Another important fact to be noted is that the dielectric loss increases with increasing temperature [3-5].

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