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Research Article

Electrical Properties of Ulexite by Dielectric Spectroscopy

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Abstract

In this work of the material investigation, electrical parameters, which are real part and imaginary part of dielectric constant versus frequency, real part and imaginary part of impedance, dissipation factor, and conductivity, in the bulk ulexite pellet is presented. Electrical properties were investigated dependent temperature and frequency with dielectric spectroscopy. Real and imaginary part of dielectric parameter properties of the ulexite were measured at frequencies from 100 Hz to 15MHz in the temperature range of 25 to 200 ºC. It was observed that AC conductivity increased with increasing frequency at room temperature and decreased with increasing frequency at higher temperatures.
Keywords: Ulexite, dielectric spectroscopy, boron, impedance, temperature.

Dielektrik Spektroskopi ile Üleksitin Elektriksel Özellikleri

Öz

Bu malzeme araştırma çalışmasında, üleksit pelletler için dielektrik sabitinin frekansa karşı reel ve sanal kısmı, empedans, kayıp faktörü, iletkenliğin reel kısmı ve sanal kısmı gibi elektriksel parametreler sunulmuştur. Elektriksel özellikler dielektrik spektroskopi ile sıcaklığa ve frekansa bağlı olarak incelenmiştir. Üleksitin dielektrik parametre özelliklerinin gerçek ve sanal kısımları, 25-200 ºC sıcaklık aralığında 100 Hz-15 MHz frekans aralığında ölçülmüştür. AC iletkenliğin oda sıcaklığında artan frekansla arttığı, daha yüksek sıcaklıklarda ise artan frekansla azaldığı gözlemlenmiştir.
Anahtar Kelimeler: Üleksit, dielektrik spektroskopi, bor, empedans, sıcaklık.

1. Introduction

Boron is one of the most important materials used in a wide range of fields such as energy sector, cleaning sector, textile type fiberglass, health and agriculture (Meydan, 2019; D. M. Schubert, 2003). Turkey has the world's largest boron reserves, so Turkey is one of the world's leading producers of boron minerals (Anaç & Tamzok, 2007; Kar, Şen, & Demirbaş, 2006). 230 different boron minerals are natural compounds containing boron oxide in different proportions (Yilmaz, 2007). Boron concentrates are either directly used in an application area or processed into a more refined boron product (Mergen & Demirhan, 2009). Boron compounds such as colemanite, tincal and ulexite are attractive materials many application areas due to their great properties (Bideci, 2016; Elçiçek & Kocakerim, 2018). Ulexite which is a mineral-rich type of boron plays an important role in heat and sound insulation, glass, ceramics and

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fertilizer industries, boric acid and borax production (Cicek, 2013; D. Schubert, 2000).

It is seen from the previous studies that boron ore have been used as an alternative radiation protective material by using with cement or concrete (Akkurt, Akyildirim, Mavi, Kilincarslan, & Basyigit, 2010; Maslehuddin, Naqvi, Ibrahim, & Kalakada, 2013). They suggested that concrete containing high percentage of boric acid and concentrated colemanite would be an effective material to protect against ionizing radiation (Aghamiri et al., 2014). Ulexite pellets can be characterized using impedance spectroscopy with the idea that they can be used in many industrial applications that require electrostatic dissipation or electromagnetic shielding (Okutan, Yalçın, et al., 2014). Characterization by impedance spectroscopy is important due to the possibility of correlating the dielectric properties and the microstructure of the system (Okutan, Yalçın, et al., 2014). Additionally, materials such as concrete, colemanite, ulexite with a high imaginer dielectric constant have high energy loss as well as high absorbency (Zhang, Wang, Wei, Guo, & Cao, 2013). Characterization by impedance spectroscopy can be used to establish structures and defects and understand origin of dielectric losses in solids. In this study dielectric properties of ulexite which is a boron mineral was investigated by impedance spectroscopy.

2. Material and Methods

The materials used in the study were collected from Eti Holding Borax and Acid Factories in Kütahya, Turkey. Ulexite, which has a specific gravity of 1.95-2 gr/cm^3, hardness (mohs) of 2.5 according to the Mohs Hardness Scale. Ulexite is boron mineral, which was crushed and grained by mechanical ways. The ulexite sample had a crushable structure and completely sieved in a 45 µm sieve. Following the material preparation processes, the sample which has 0.5 g weight, was pelletized with a hydraulic press under a pressure of 10 tons. The thickness and diameter of the prepared pellets were found to be 1 mm and 12 mm, respectively. The surface morphology of the sample was analyzed using SEM (Carl Zeiss 300VP SEM).

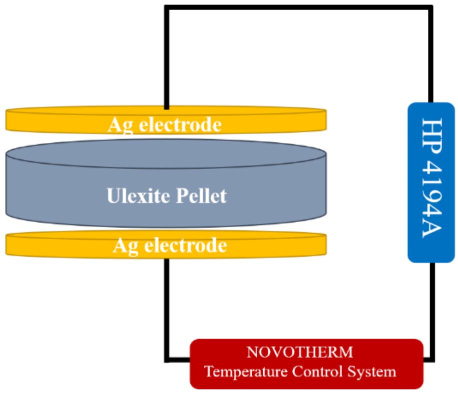


Figure 1. Schematic illustration of ulexite pellet and experimental setup.

In order to determine the electrical properties of the ulexite pellets, the ulexite samples which were sandwiched between two Ag electrodes were measured with dielectric spectroscopy method by applying different frequencies at different temperatures (Figure 1). For this dielectric spectroscopy measurements, HP 4194A impedance analyzer was used. It has not been done annealing process. Temperature dependent complex impedance was measured at logarithmic frequencies between 100 Hz and 15 MHz. For temperature control, the NOVOTHERM Temperature Control System (NTCS) which is enabled for monitoring the temperature in the 25-200 ºC range. The crystal structure of ulexite was investigated by X-ray diffraction method using Panalytical Empyrean XRD system. The presence of materials obtained from the X-Ray spectrum by EDX has also been confirmed.

3. Results and Discussion

The surface roughness reflected in the height of profiles of the images and the surface distributions of the ulexite pellet was recorded by Carl Zeiss 300VP scanning electron micrograph.

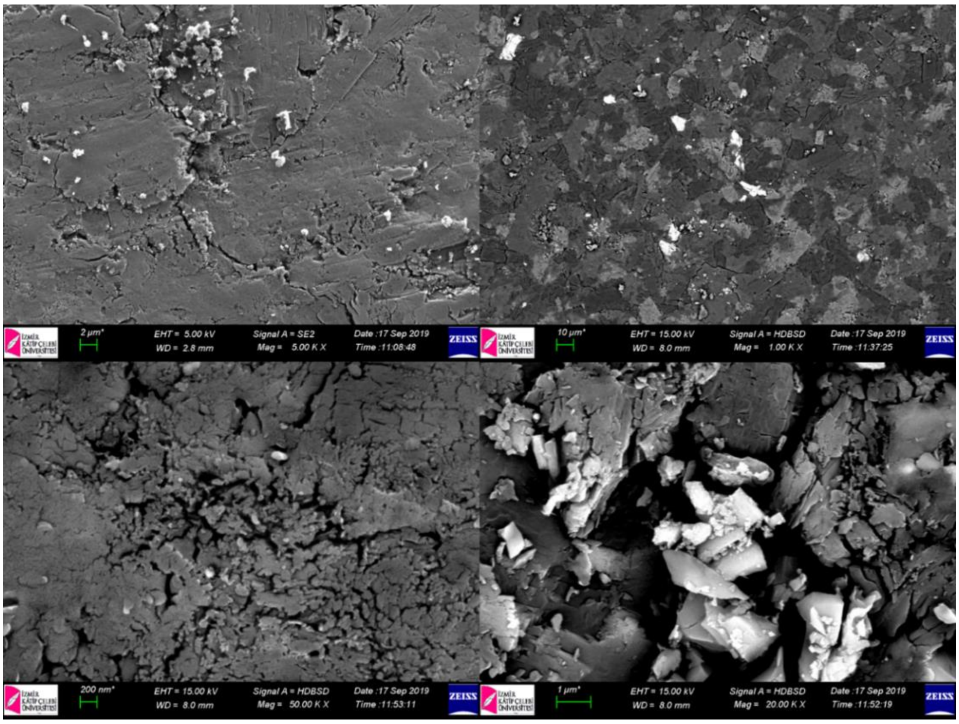
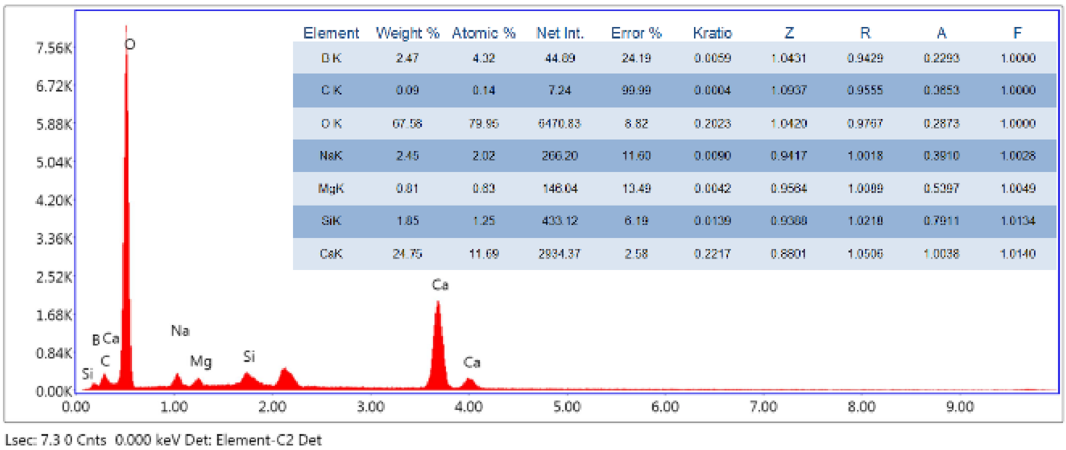


Figure 2. The scanning electron micrograph of the ulexite pellet sample

The scanning electron micrograph of the ulexite sample at magnification of 1,2, 10 µm and 200 nm are shown in figure 2. The other parameters of surface morphology of the ulexite are also presented on figure 2. SEM images indicate that morphology of ulexite pellet surface has irregular.



Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	R	A	F
B K	2.47	4.32	44.69	24.19	0.0059	1.0431	0.9429	0.2293	1.0000
C K	0.09	0.14	7.24	99.99	0.0004	1.0937	0.9555	0.3853	1.0000
O K	67.58	79.95	6470.83	8.82	0.2023	1.0420	0.9767	0.2873	1.0000
NaK	2.45	2.02	266.20	11.60	0.0090	0.9417	1.0018	0.3910	1.0028
MgK	0.81	0.63	146.04	13.49	0.0042	0.9584	1.0099	0.5397	1.0049
SiK	1.85	1.25	433.12	6.19	0.0139	0.9388	1.0218	0.7911	1.0134
CaK	24.75	11.69	2934.37	2.58	0.2217	0.8801	1.0506	1.0038	1.0140

Figure 3. EDX spectrum of ulexite pellet and data of EDX measurements of sample is given the inset table.

The grains are inhomogeneously distributed throughout the surface of the sample. Micrographs exhibit that the grain size of the pellet sample was found to be about 1.5 µm.

The elemental analysis (EDX) spectrum ulexite pellet illustrated in figure 3, indicate the presence of B, C, O, Na, Mg, Si, Ca, in the sample.

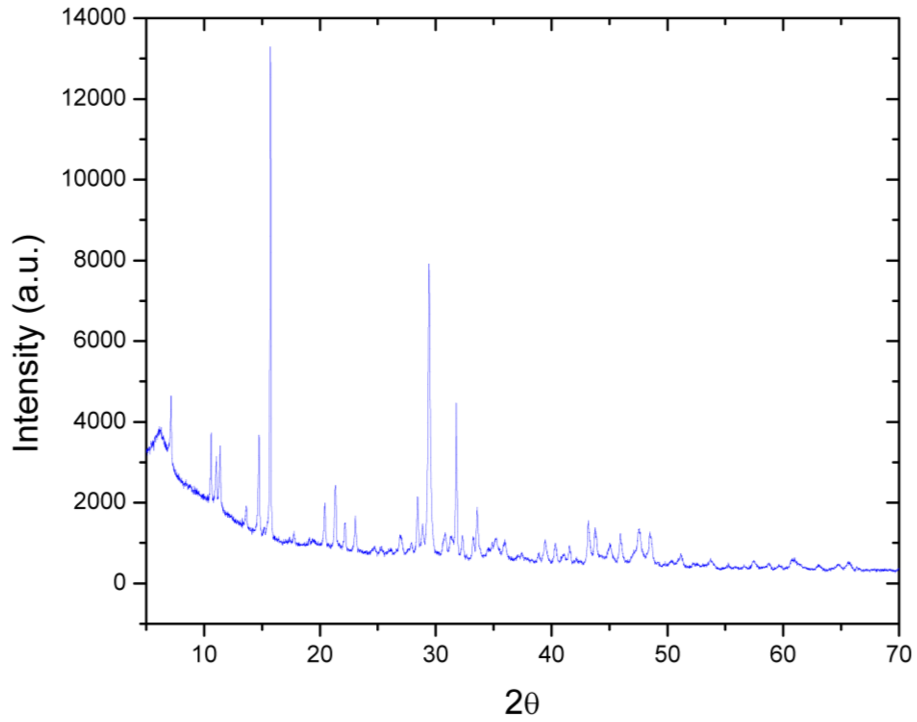


Figure 4. XRD patterns of ulexite

The room temperature X-ray diffraction (XRD) pattern of ulexite ($NaCaB_5O_9.8H_2O$) is shown in figure 4. The XRD pattern (i.e., peak position and intensity) clearly shows the formation of a ulexite obtained from Kestelek boron facilities. Using 2θ, interplaner spacing (d) of each peak was obtained.

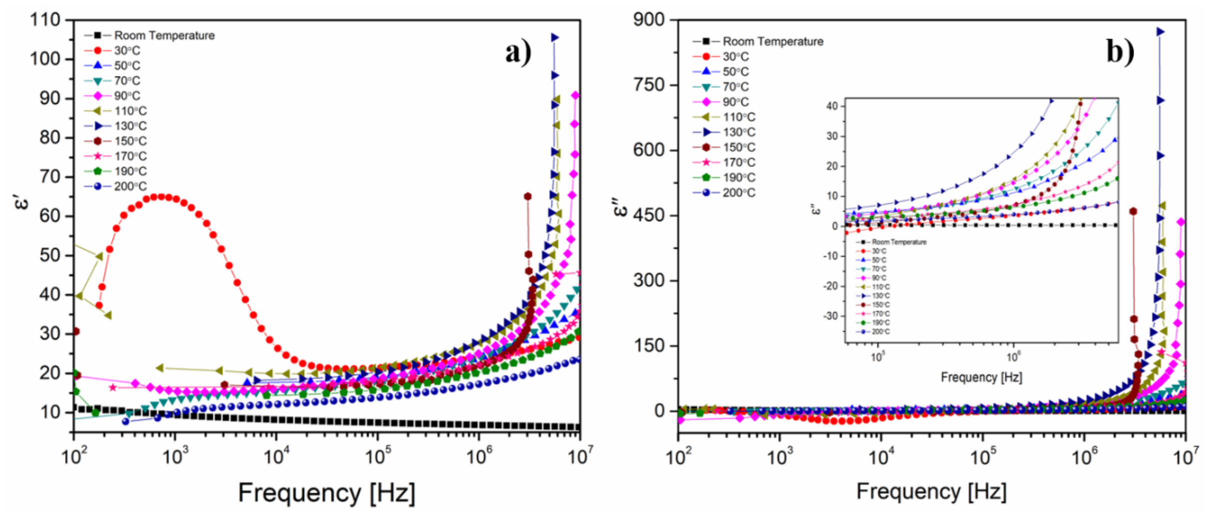


Figure 5. Variation of the (a) real (ε′) and (b) imaginary (ε″) part of complex dielectric constant as a function of frequency at different temperature for ulexite.

Variation of the real (ε′) (Fig. 5a) and imaginer (ε″) (Fig. 5a) part of dielectric constant as a function of frequency at different temperature for ulexite. Dielectric relaxation peak indicates a phase transition and the presence of temperature dispersion and the tangent loss supports the phenomena of diffuse phase transition (Okutan, Kavanoz, et al., 2014).

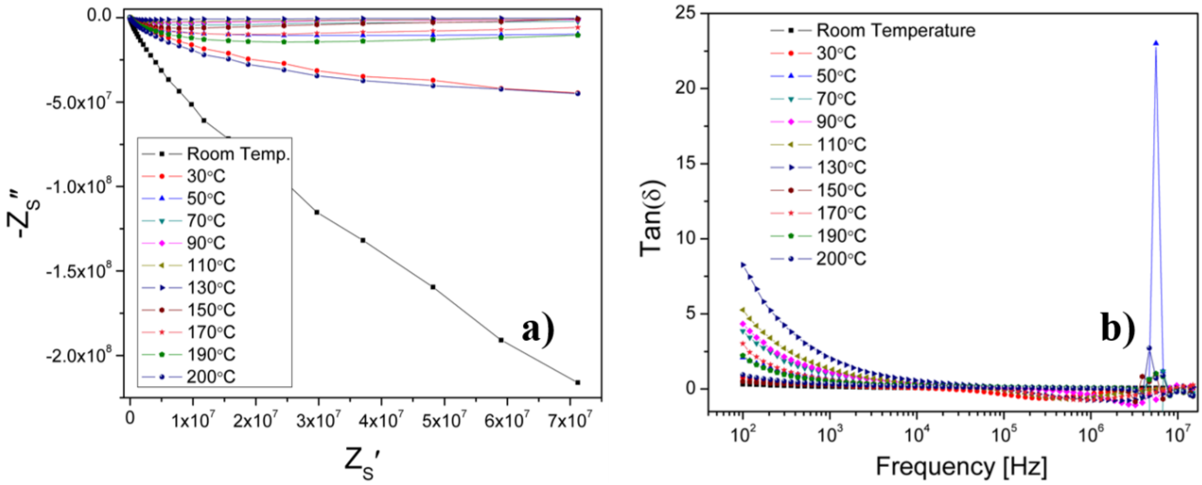


Figure 6. (a) The Cole- Cole plots of the ulexite pellet **(b)** shows loss tangent (δ) as a function of frequency at room temperature for ulexite.

The Cole- Cole plots of the ulexite pellet was shown in figure 6 (a). Figure 6(b) shows loss tangent (δ) as a function of frequency at room temperature for ulexite. This behavior occurs because, at low frequencies, dipoles easily respond and align with the applied alternating electric field but as frequency increases diploes cannot cope up with the pace of fast changing electric field and therefore dielectric constant decreases and become saturated at further higher frequencies.

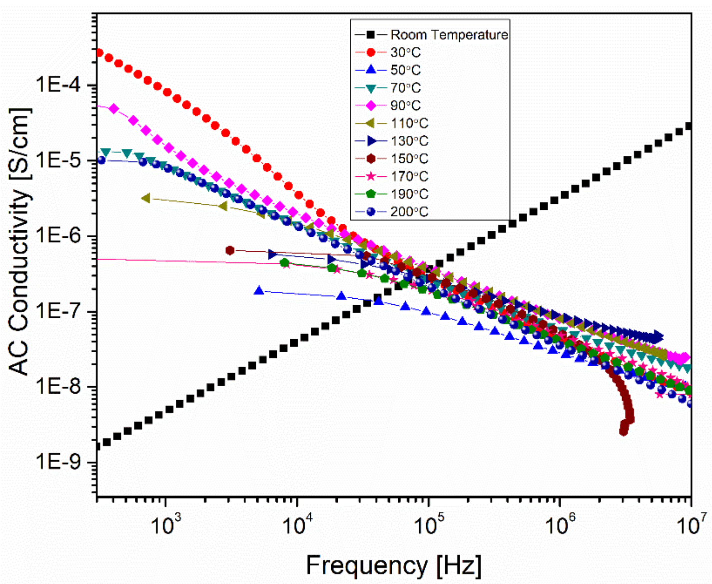


Figure 7. AC Conductivity versus frequency dependent temperature for ulexite pellet

But in this work, there is anomalies. Dielectric constant values have increased with increased frequency. While the real part of the dielectric permittivity (ε′) has the same values because of no charge accumulation at the interface, which can be explained by the ion diffusion mechanism at higher frequencies; the charges get accumulated at the interfacial region at lower frequencies (Bazan, Duffy, Ingram, & Mallace, 1996; Sidebottom, Green, & Brow, 1995). Additionally, AC Conductivity versus frequency dependent temperature for ulexite pellet was shown in figure 7.

4. Conclusion

In this paper, an effort has been made to determine the dielectric properties of ulexite. Dielectric properties of ulexite which is boron mineral was investigated by impedance spectroscopy. Electrical properties were investigated dependent temperature and frequency with dielectric spectroscopy. Real and imaginary part of dielectric parameter properties of the ulexite were measured at frequencies from 100 Hz to 15MHz in the temperature range of 25 to 200 °C. It was observed that AC conductivity increased with increasing frequency at room temperature and AC conductivity decreased with increasing frequency at higher temperatures for ulexite. Additionally, the dielectric losses have increased with increasing frequency. This increase is more rapid in the dispersion region. At higher frequency, dielectric constant has increased to 150 ºC and then began to decline. Anomalies, near 170, 190 and 200 ºC which are particularly well seen in the temperature dependence of (Fig. 5). The temperatures 30 ºC are close to the temperatures of anomalies in water (ice–water first-order phase transition and anomalies in the compressibility and heat capacity of water (Gavrilova, Lotonov, & Kornilova, 2008). The likely reason for this is that the structure of ulexite contains hydrogen bonds, the same as in the structure of water.

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