Scientific Review
ISSN(e): 2412-2599, ISSN(p): 2413-8835
Vol. 11, Issue. 1, pp: 1-9, 2025
URL: https://arpgweb.com/journal/journal/10
DOI: https://doi.org/10.32861/sr.111.1.9

Academic Research Publishing Group

Original Research Open Access

Multi-ferroic Properties of Ammonium Sulphate Gotten from Ammonium Metavanadate and Sulphuric Acid

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Article History
Received: 15 December, 2024
Revised: 21 February, 2025
Accepted: 8 March, 2025
Published: 12 March, 2025

Abstract

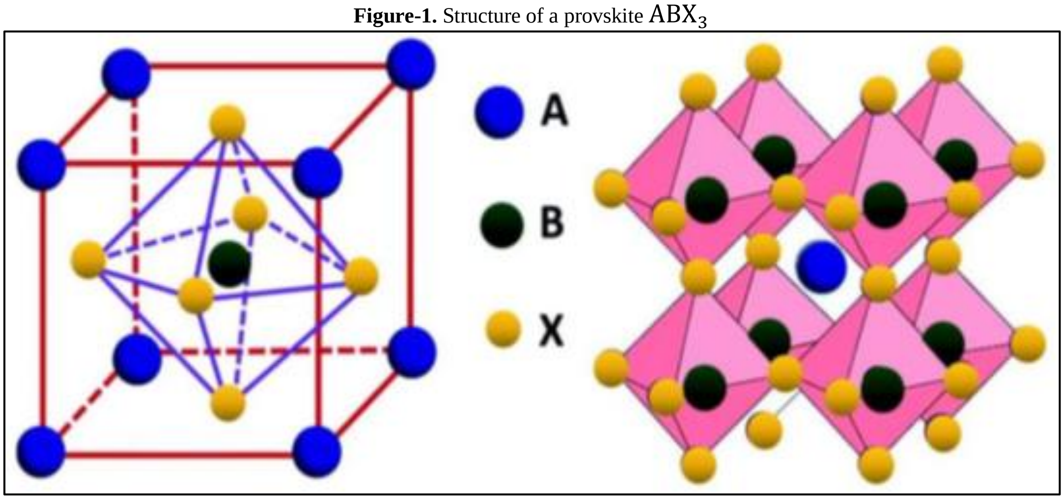
There is today a growing interest for ferroic materials. Some of them are ferroelectric, ferromagnetic or flexoelectric. Some materials are multiferroic, being both ferroelectric and ferromagnetic Multiferroic materials are rare. The present study showed that the ammonium sulphate, gotten from ammonium metavanadate and sulphuric acid is ferroelectric and ferromagnetic. There are electric dipoles at the surface and also inside the material. The electric signal was recorded and visualized. The Fourier transform carried out shown, in the angular frequency spectrum, the presence of four main lines. The *drawing* of the profiles of the two highest lines showed that they have doubly peaked profiles.

Keywords: Multiferroic materials; Ferroelectricity; Ferromagnetism; Ammonium sulphate; Cation frequency.

1. Introduction

There is today a growing interest for ferroic materials. Some of them are ferroelectric, ferromagnetic or flexoelectric. Some materials are multiferroic, being both ferroelectric and ferromagnetic. According to literature, multiferroic materials are rare (Kreisel , J. et al , 2008). Ferroic materials have several applications. An example is PZT (PbZr1-xTixO3) for which a coupling between deformation and electric polarisation yields the piezoelectric effect. The perovskites ABX_3 are ferroelectric materials, which are used in photovoltaic conversion of solar energy (Soro, D. et al , 2017), (Soro, D. et al , 2018). Another property is ferroelasticity found in the perovskite SrTiO3. The structure of an example of perovskite is shown in figure 1 (Brittman, S. et al, 2015), (Lang, L. et al, 2013).

Figure-1. Structure of a provskite ABX_3



In figure 1 A and B are the cations

The present study deals with a kind of multiferroic material, found when we were looking for the vanadium V, during experiments for the study of a vanadium Redox Battery.

2. Materials and Methods

In literature, ammonium sulphate is gotten by pulverising sulphuric acid in ammoniac gas according to the following reaction

$$2NH_3 + H_2SO_4 \rightarrow (NH_4)_2SO_4 \qquad (1)$$

This ammonium sulphate may have the appearance of granules or crystals. Its density is reported to lie within the range 1.76 -1.77 g/cm^3

For this material, no ferroic property is reported in literature.

In the present work, ammonium sulphate is obtained as a precipitation in the reaction between ammonium metavanadate and sulphuric acid, as shown in equation (2)

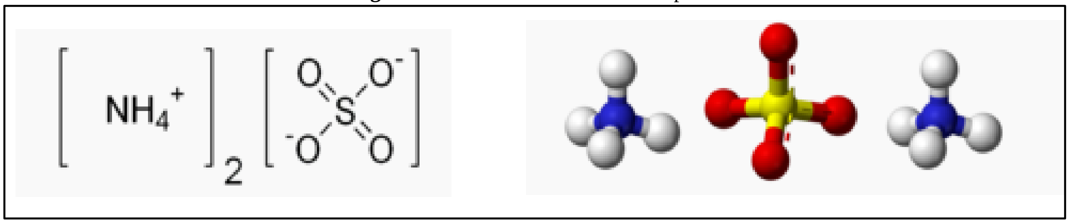
$$2\,NH_4VO_3 + H_2SO_4 \rightarrow V_2O_5 + (NH_4)_2SO_4 + H_2O \qquad (2)$$

There is a precipitation of ammonium sulphate while the yellow vanadium pentoxide remains in the solution.

The precipitation was poured out on a sheet of white paper. At this stage, it is brown. Then it is dried at room temperature during several weeks. After drying, it becomes rather black. The density was measured. It was found $1.75 \pm 0.02\ g/cm^3$

According to literature the structure of ammonium sulphate is as indicated in figure 2

Figure-2. Structure of ammonium sulphate



In a first experiment, the two terminals of a multimeter were connected to one sample. A quickly varying voltage was observed. An electric current was also observed

This electric property led us to verify the presence of charges at the surface of the material. Small pieces of white paper were cut. Figure 3 shows that they are attracted by samples of ammonium sulphate

Figure-3. Attraction of pieces of paper by a sample of ammonium sulphate

Then the magnetic properties of ammonium sulphate was checked. Figure 4 shows that a sample of ammonium sulphate is attracted by a screw.

Figure-4. Attraction of a sample of ammonium sulphate by a screw

This magnetic property led us to check the interaction between a sample of ammonium sulphate and a magnet. Figure 5 shows that it is of course attracted by a magnet.

Figure-5. Attraction of a sample of ammonium sulphate by a magnet

Now, it was necessary to visualize the electric signal seen when the terminals of a multimeter are put on a sample of the material. The voltage varies quickly, so that it was necessary to amplify the electric signal, store it and then visualise it on a computer using a microcontroller which converts the analogue signal into a digital signal.

A non-inverting operational amplifier LM 124 J was used. Its voltage gain G is expressed as

$$G = \frac{V_{out}}{V_{in}} = 1 + \frac{R_f}{R_1} \quad (3)$$

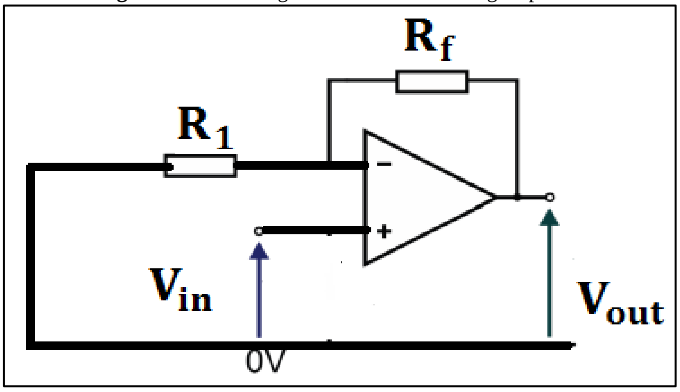
The following values were chosen for the resistances:

$R_f = 3\ k\Omega$; $R_1 = 1\ k\Omega$

The voltage gain is therefore G = 4

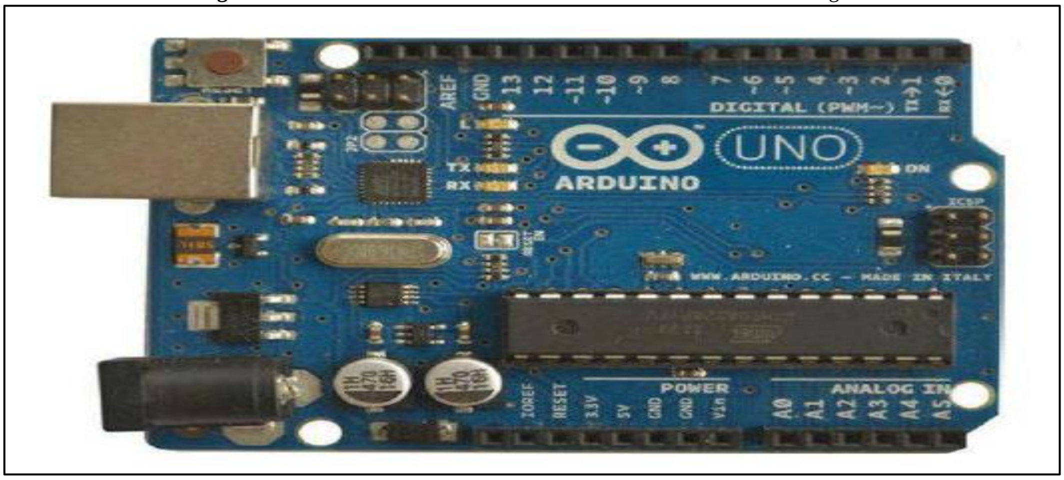
Figure 6 shows the circuit diagram of the amplifier

Figure-6. Circuit diagram of the non-inverting amplifier



For the visualization of the electric signal after its amplification, the microcontroller used is shown in figure 7.

Figure-7. The ARDUINO microcontroller used to visualize the electric signal



The analogue signal is converted to a digital signal and connected to a microcomputer (PC). The whole circuit is sketched in figure 8.

Figure-8. Whole circuit for the visualization of the electric signal by a PC.

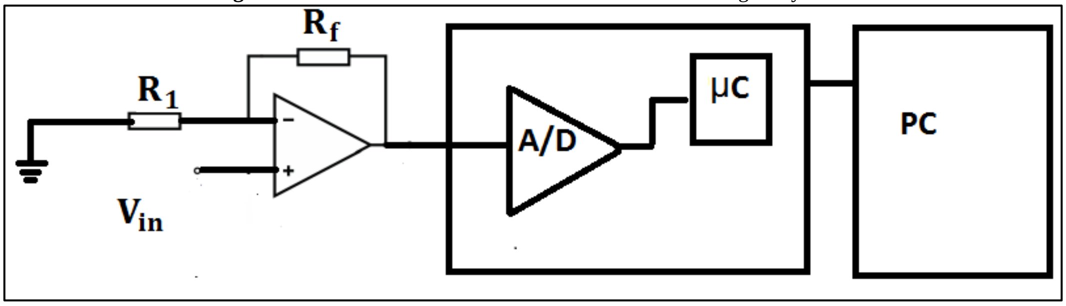
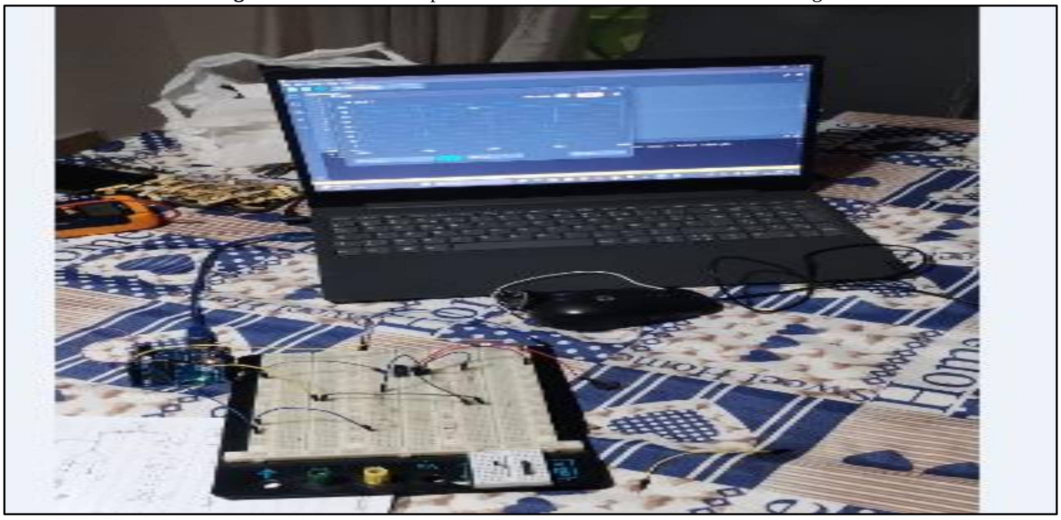


Figure 9 shows the practical set-up of the visualization circuit

Figure-9. Practical set-up of the visualization circuit of the electric signal



3. Results and Discussions

The first experiments showed the apparition of voltage when the two terminals of a multimeter are connected to one sample of ammonium sulphate. This voltage shows the presence of electric charges at the surface of the material. The attraction of small pieces of paper by the material reinforce this affirmation. The charges of the material are responsible for an electric field, which provokes a polarisation of the piece of paper, resulting in an electric attraction between the sample and the piece of paper.

It was necessary to know whether or not there are also charges inside the product. For that, samples of different volumes were experimented. For each sample, the maximum voltage observed, and the maximum intensity measured were noticed. The results are shown in table 1

Table-1. Maximum voltage and maximum intensity measured for various volumes of sample.

Volume (mm^3)	Maximum voltage (V)	Maximum intensity (mA)
24	0.14	0.14
40	0.20	0.25
154	0.8	0.33

Obviously, the voltage increases strongly when the volume of the sample is increased. There is also an increasing of the intensity, but at a slower rate. This experiment shows the presence of charges inside the sample, as well as at its surface.

An electric dipole consists of a positive charge (+e) and a negative charge (-e), which are at distance x(t) from each other. A dipole moment p is defined as p = e. x(t)

Let a single charge e be considered in harmonic motion. Electromagnetic theory shows that it will produce an electric field and a magnetic field. (Garbuny, M., 1965).The electric field E is proportional to $\ddot{p}(t) = e.\ddot{x}(t)$ in its first term. Other terms of the electric field involve $\dot{p}(t) = e.\dot{x}(t)$ and x(t)

As for the magnetic field H, it is the addition of two terms: the first one is proportional to $\ddot{\mathbf{p}}(\mathbf{t}) = \mathbf{e}.\ddot{\mathbf{x}}(\mathbf{t})$, while the second term is proportional to $\dot{p}(t) = e.\dot{x}(t)$

Let a charge be considered, at distance $\vec{\mathbf{R}}$ from a point O. When it is assumed that there are several dipole moments in a material, the multipole moment is defined as

$$\vec{X} = \sum_k e_k . \vec{x}_k(t) \qquad (4)$$

For $R \ll \lambda$ (where λ is the wavelength), only the first terms are considered, so that we have

$$\vec{E} = \frac{1}{R^3c^2}[\vec{R} \times (\vec{R} \times \ddot{\vec{X}})] \qquad (5)$$

$$\vec{H} = \frac{1}{R^2c^2}[\ddot{\vec{X}} \times \vec{R}] \qquad (6)$$

When it is assumed that $\vec{\mathbf{X}}$ represents a harmonic oscillator multipole moment at time t' = t-R/c, $\vec{\mathbf{X}}$ may be defined as

$$\vec{X} = \vec{X}_0 e^{i\omega(t-\frac{R}{c})} \qquad (7)$$

Then, from equations (5) and (6), one gets

$$\vec{E} = \frac{\omega^2}{R^3c^2} e^{i\omega(t-\frac{R}{c})}[\vec{R} \times (\vec{X}_0 \times \vec{R})] \qquad (8)$$

$$\vec{H} = \frac{\boldsymbol{\omega}^2}{R^2c^2} e^{i\omega(t-\frac{R}{c})}[\vec{R} \times \overrightarrow{\vec{X}_0}] \qquad (9)$$

Moreover, the electric field E is the electromotive force (e.m.f.) V gradient, so that we have

$$E = \frac{V}{l} \qquad (10)$$

where l is a length of the material

Equations (8), (9) and (10) show that the frequency of the e.m.f. V is also the frequency of E and H. Consequently, after the recording of the amplified signal, it was necessary to make a Fourier transform of that signal, which shows the oscillation frequencies of the dipoles present in the material.

According to the Fourier theorem a periodic function f (t) with period T = 2π/ω can be expressed as

$$f(t) = \frac{a_0}{2} + \sum_{k=1}^{n} a_k \cos k\omega_k t + \sum_{k=1}^{n} b_k \sin k\omega_k t \qquad (11)$$

That gives in complex notation

$$f(t) = \sum_{n\epsilon Z} c_n e^{i\omega_n t} \qquad (12)$$

Where c_n is expressed as

$$c_n = \frac{a_n - ib_n}{2} \qquad (13)$$

From those relations, the Fourier transform is defined as

$$F(\omega) = (2\pi)^{-1/2} \int_{-\infty}^{+\infty} f(t).e^{-i\omega_n t}\, dt \qquad (14)$$

A signal f (t) can also be calculated as the inverse Fourier transform of F (ω)

Hence, when the temporal representation f (t) of a signal is known, its frequency representation F (ω) can be calculated by equation (14), for each angular frequency

ω= 2πν, where ν is the frequency.

The electric and magnetic properties observed are the consequences of charge motions. As ammonium sulphate has two components $(NH_4)_2$ and SO_4 , its structure shown on figure 2 shows that the two (NH_4) may oscillate as cations, one on the right and the second on the left of the second component which is SO_4

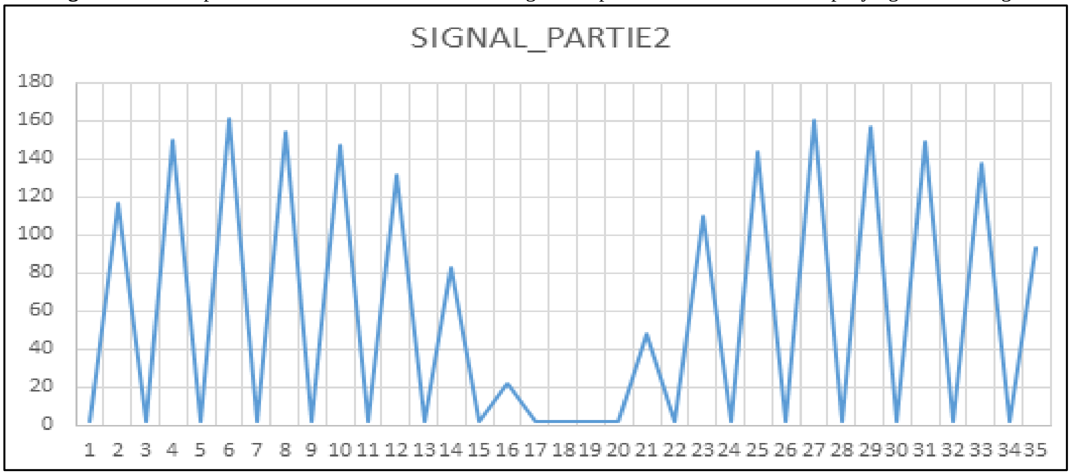
Each cation may have its own motion, resulting in a non-null multipole moment with different oscillation frequencies.

Another hypothesis for the dipole moment production in the material could be an oscillation motion of SO_4 between the two (NH_4) .

But this hypothesis is less likely than the first one, because of the heavier mass of SO_4, if compared with the mass of $(NH_4)_2$. For different samples, the recording of the electric voltage was carried out, according to the circuit shown in figure 8 and figure 9.

An example of recorded signal is given in figure 10. The amplified signal is recorded every 0.01 sec. The figure shows fast varying signals.

Figure-10. Example 1 of visualization of the electric signal temporal variation after the amplifying of the voltage



The Fourier analysis of the signal shown in figure 10 is given in figure 11.

Figure-11. Fourier analysis result regarding the example 1 recording.

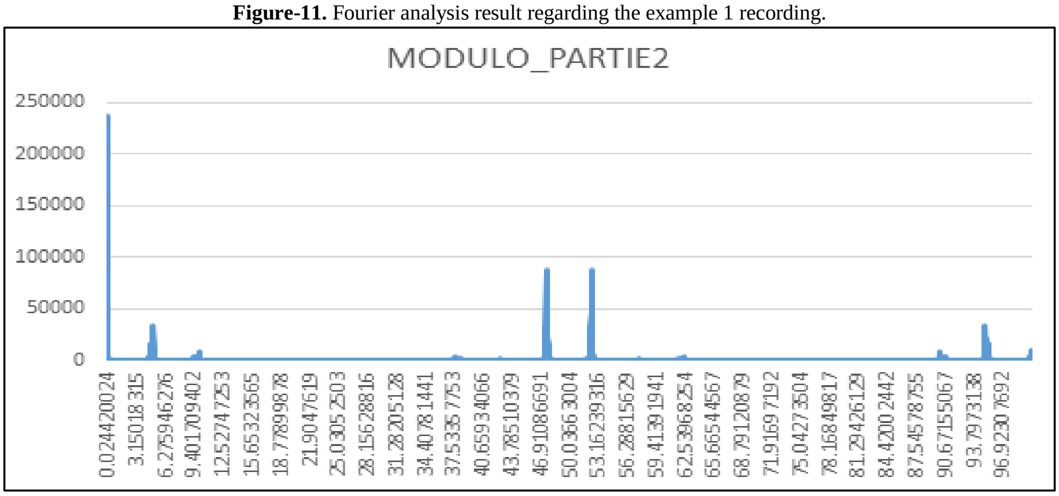


Figure 11 shows, in the angular frequency spectrum, four major lines. Their angular frequencies are the followings. For line 1: $\omega_1 = 5.006\ s^{-1}$; for line 2: $\omega_2 = 47.546\ s^{-1}$; for line 3: $\omega_3 = 52.527\ s^{-1}$; for line 4: $\omega_4 = 95.067\ s^{-1}$

Every line has a profile. The shapes of line 2 and line 3, the highest lines, are given below, in figures 12 and 13

Figure-12. Shape of line 2

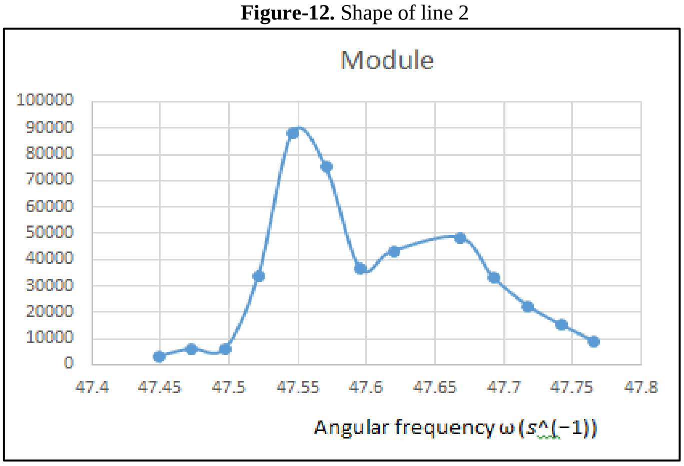
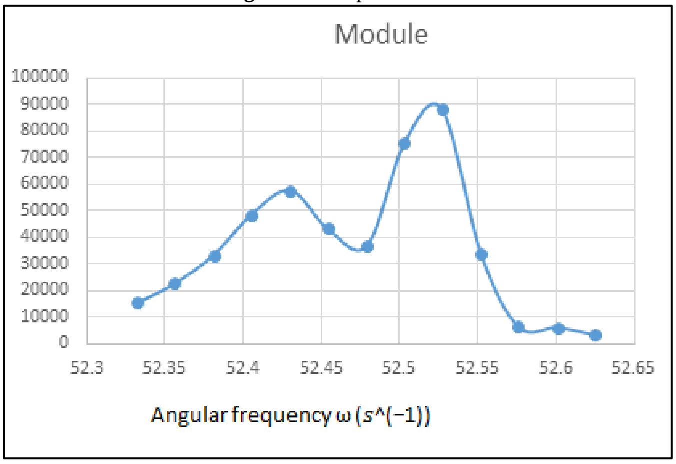
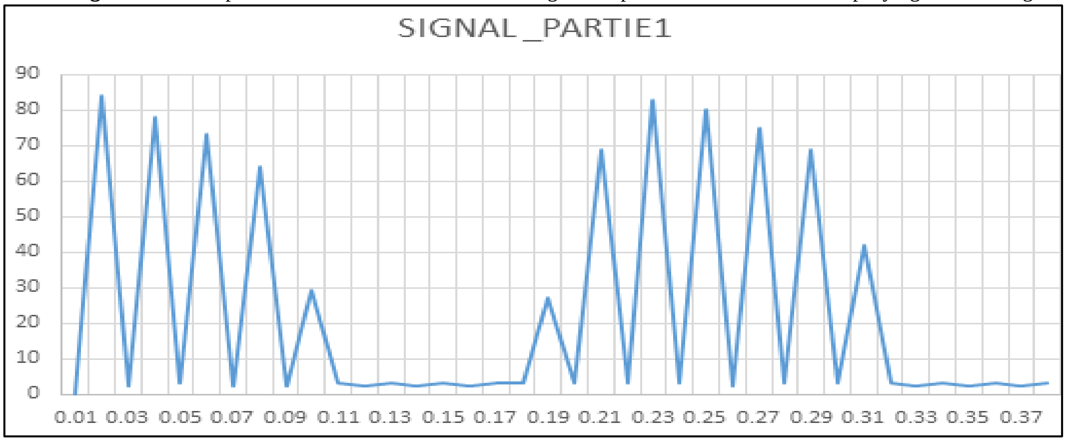


Figure-13. Shape of line 3



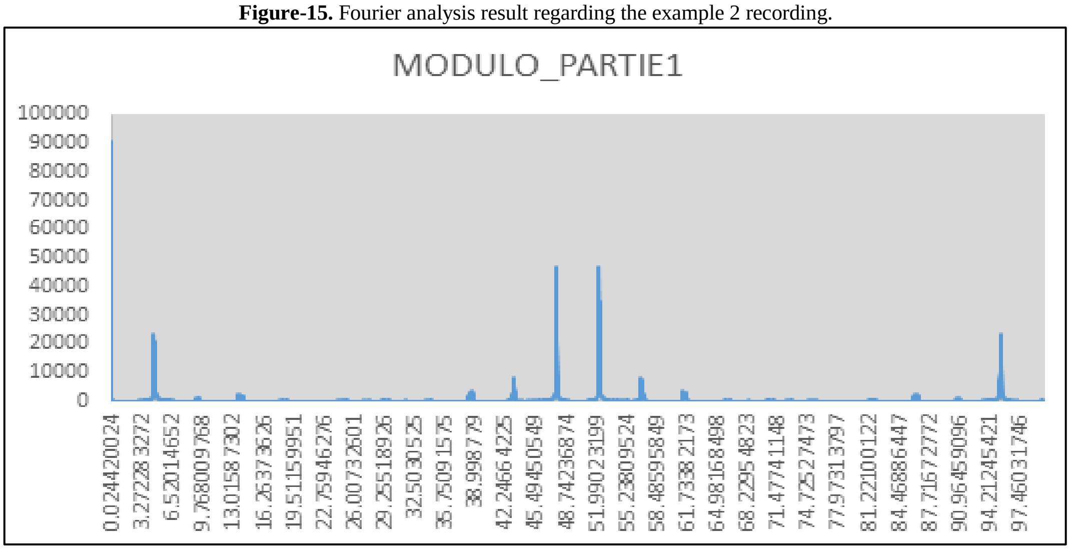
Another example of recorded signal is given in figure 14. The figure shows also fast varying signals.

Figure-14. Example 2 of visualization of the electric signal temporal variation after the amplifying of the voltage



The Fourier analysis of this second signal is given in figure 15

Figure-15. Fourier analysis result regarding the example 2 recording.



Here again, in the angular frequency spectrum, four major lines are found. Their angular frequencies are the followings. For line 1: $\omega_1 = 4.566\ s^{-1}$; for line 2: $\omega_2 = 47.765\ s^{-1}$; for line 3: $\omega_3 = 52.308\ s^{-1}$; for line 4: $\omega_4 = 95.506\ s^{-1}$

As for the first experiment, the shapes of line 2 and line 3, the highest lines, were drawn; they are given below, in figures 16 and 17

Figure-16. Profile of line 2, for the second experiment

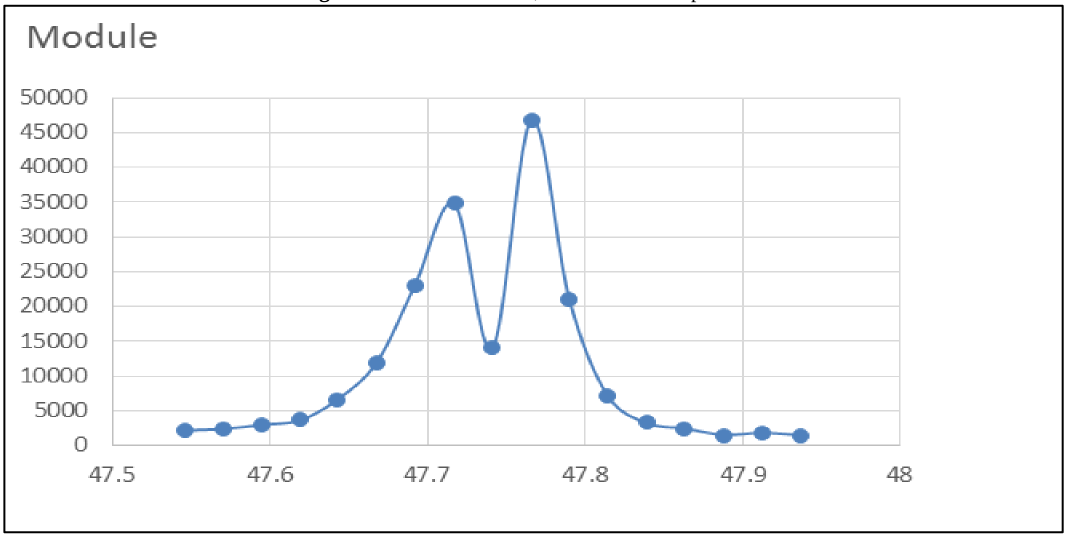
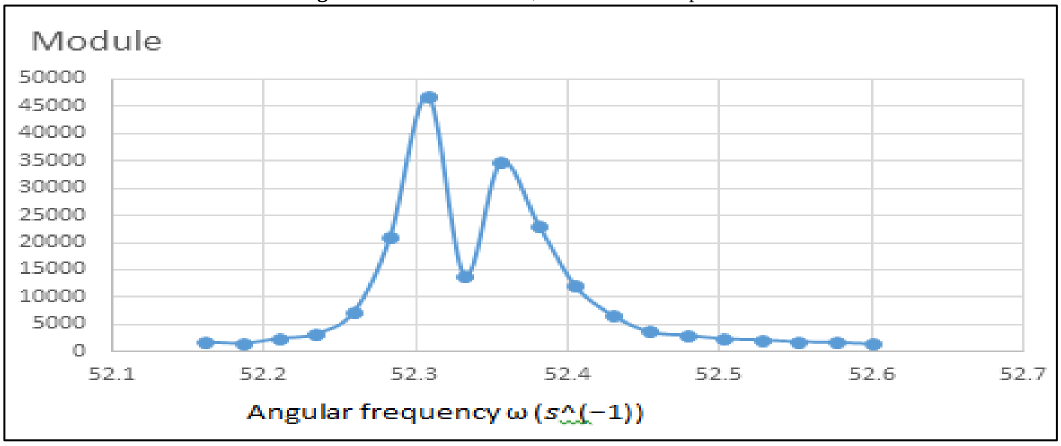


Figure-17. Profile of line 3, for the second experiment



It is noteworthy to mention that, for the two experiments, line 2 and line 3 have doubly peaked profiles. For each line, the profile is not symmetric. It is also worth noting that, in the second experiment, the profiles are sharper. On the whole, for the two different experiences, the same lines are observed.

This study is relative to the electric signal. It is also interesting to study the magnetic signal. A sensor can transform the magnetic signal into an electric signal that can be recorded and analysed. A study should also be made about the cation motions which are responsible for the signal and the frequency spectrum observed. That will be the matter of a next study.

4. Conclusion

The present study showed that the ammonium sulphate, gotten from ammonium metavanadate and sulphuric acid is ferroelectric and ferromagnetic. There are electric dipoles at the surface and also inside the material. The electric signal was recorded and visualized. The Fourier transform carried out shown, in the angular frequency spectrum, the presence of four main lines. The drawing of the profiles of the two highest lines showed that they have doubly peaked profiles. As perspective, the magnetic signal will be recorded and a Fourier transform of this signal will be made. A study will be also made about the cation motions involved in the frequency spectrum observed.

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