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Ferroelectric Material Triglycine Sulphate (TGS): Properties, Synthesis, and Applications

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***Abstract:** This comprehensive review examines the ferroelectric material triglycine sulphate (TGS), focusing on its properties, synthesis methods, and applications up to 2021. TGS has garnered significant attention in the field of ferroelectrics due to its exceptional pyroelectric and piezoelectric properties. This paper provides an in-depth analysis of TGS's crystal structure, ferroelectric characteristics, and temperature-dependent dielectric properties. Various synthesis techniques and characterization methods are discussed, along with recent advancements in TGS research. The review also explores the advantages, challenges, and potential applications of TGS in diverse fields such as infrared detection, electro-optic devices, and energy harvesting. Finally, future research directions are proposed to further enhance the understanding and utilization of this remarkable ferroelectric material.*

***Keywords:** Triglycine Sulphate, Ferroelectric Materials, Pyroelectric, Piezoelectric, Crystal Growth, Dielectric Properties.*

1. INTRODUCTION

Overview of Ferroelectric Materials

Ferroelectric materials have been at the forefront of materials science research due to their unique ability to exhibit spontaneous electric polarization that can be reversed by an external electric field. These materials display a wide range of functional properties, including piezoelectricity, pyroelectricity, and high dielectric constants, making them invaluable in various technological applications [5]. The discovery of ferroelectricity in 1920 by Joseph Valasek in Rochelle salt opened up a new field of study, leading to the identification and development of numerous ferroelectric materials over the past century.

Ferroelectric materials may be defined as materials which can exhibit a spontaneous electric polarization in the absence of an external electric field. This state of polarization can be reversed by application of an electric field of sufficient magnitude termed as coercive field.

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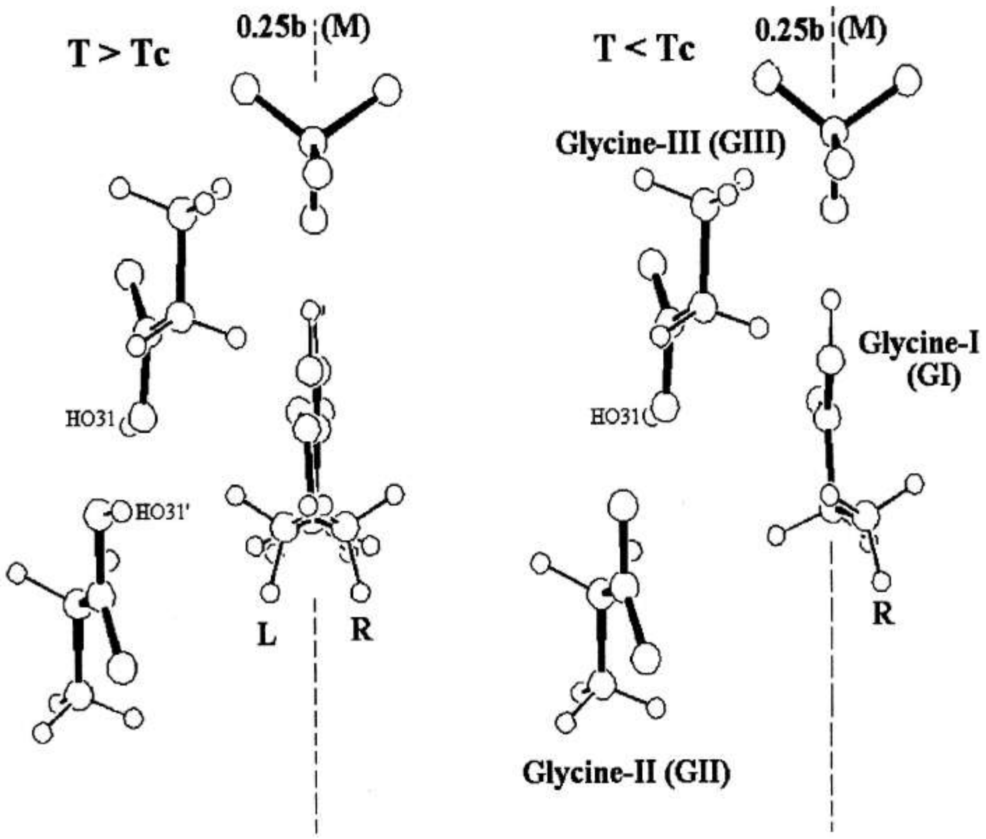
Polarization is the measure of dipole moment per unit volume in ferroelectric materials and it has a relation with the applied electric field which is depicted by hysteresis loop [3]. These materials also have a Curie temperature that is a temperature at which the materials change from the ferroelectric state to the paraelectric state and lose the spontaneous polarization.

Brief Introduction to Triglycine Sulphate (TGS)

Triglycine sulphate (NH2CH2COOH)3·H2SO4, TGS for short, is a typical organic ferroelectric material which has been investigated intensively since it was found in the 1950s. TGS is a crystal which is an isomorphic modification of tris-methanoammonium chloride and belongs to the monoclinic system; it has a second order phase transition at its Curie temperature of about 49°C. This phase transition involves the transition from the ferroelectric phase with the space group of P21 to the paraelectric phase with the space group of P21/m with a drastic change of physical properties [1].

TGS has become an important material in the field of ferroelectrics because of its high pyroelectric and piezoelectric coefficients, low dielectric loss and room temperature ferroelectricity. These characteristics make TGS especially suitable for application in the infrared detectors, electro-optical devices and transducers. The material has a comparatively low Curie temperature, which enables one to easily study the material's ferroelectric-paraelectric phase transition thus making it a suitable material for fundamental studies of ferroelectricity.



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Importance of TGS in Ferroelectric Applications

The role of TGS in ferroelectric applications is due to the following characteristics: a high pyroelectric coefficient and low dielectric constant that results in a high figure of merit for pyroelectric applications. Pyroelectricity in TGS provides for the ability to transform temperature variation into electrical signals thus making it highly effective in detecting infrared radiation. This property together with its low dielectric constant give high voltage responsivity that is very important in the making of infrared detectors.

Besides, the organic nature of TGS is more beneficial than the inorganic ferroelectric material as discussed below. This material can be easily synthesized in the form of large and high optical quality single crystals by solution growth methods. This ease of crystal growth together with the fact that TGS exhibits ferroelectricity at room temperature makes TGS a very useful material in both theoretical and applied research.

2. LITERATURE REVIEW

Historical Development of TGS Research

Ferroelectric properties of TGS were first discovered by Matthias and coworkers in 1956 which is an important achievement in the field of organic ferroelectrics. This discovery attracted researchers' attention to TGS and many works were done to investigate its structural, electrical and optical properties. Substantial improvement in the growth of large, high-quality TGS crystals was achieved in the 1960s and 1970s and this opened the door for further investigations and uses [7][8].

Research has also been conducted in relation to TGS and the following studies have been found to be very informative. Hoshino et al. (1959) did a thorough work on the X-ray analysis of the crystal structure where they explained the formation of glycine molecules and the hydrogen bonds. Their work offered important information about the nature of the structure of ferroelectricity in TGS, how the glycine molecules and hydrogen bonds are involved in the process.

Key Studies and Findings on TGS

Gonzalo (1990) gave a detailed account of the ferroelectric properties of TGS together with domain configuration and switching characteristics. This work integrative much of the current information about TGS and its possibilities for use in different areas. Kay and Vousden (1949) made early work in the temperature variation of dielectric constants of TGS and the characteristic of the ferroelectric phase transition. They helped in the elucidation of the fact that the phase transition in TGS is of second order.

Fugiel and Malek (1988) studied the effects of electric field on the dielectric properties of TGS materials and this give a good understanding of the material's behaviour under the influence of an electric field. They have made a contribution to the study of the switching processes in TGS and the conditions influencing the ferroelectric behaviour.

In the course of the investigation of crystal growth, Mihaylova and Mehandjiev (1984) provided valuable information by working on the techniques of growing large TGS crystals of high quality. It enabled the growth of crystals which are ideal for use and examination through various scientific experiments.

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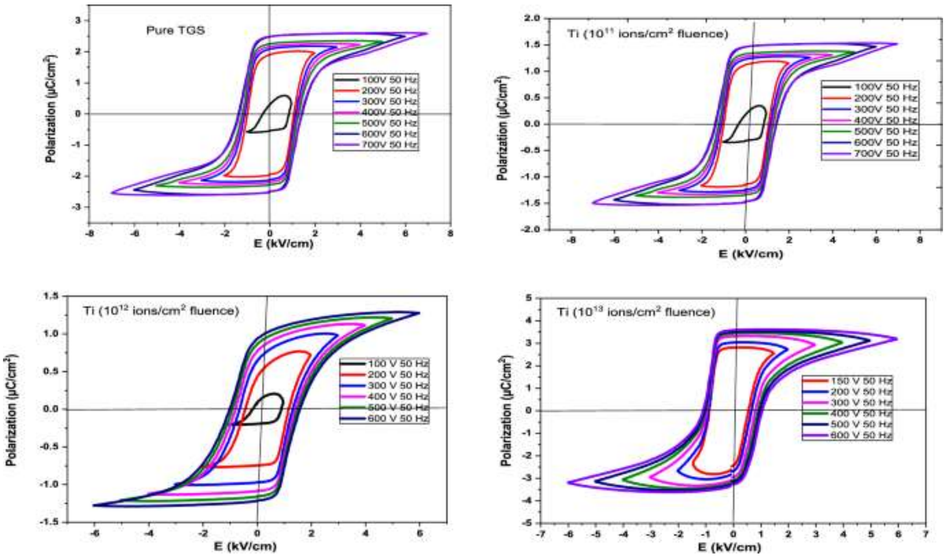

Nakamura et al. (1964) have carried out detailed investigations on the pyroelectric characteristics of TGS and have reported high pyroelectric coefficient and the possibility of using TGS for infrared detection system. These works formed the basis of the TGS based pyroelectric devices.

The subsequent works have been devoted to the improvement of TGS characteristics by doping and varying the composition. For instance, Bhalla et al. (2000) studied the influence of different dopants on the pyroelectric properties of TGS with the purpose of enhancing the material's performance in uncooled infrared detectors.

TGS has several beneficial properties when compared with other ferroelectric materials, including barium titanate $BaTiO_3$ and lead zirconate titanate PZT. This has a relatively low Curie temperature of 50°C, and is therefore ideal for applications operating at room temperature, as well as showing a higher pyroelectric coefficient. The pyroelectric coefficient of TGS is about 3[13]. This value is about 5×10^{-4} C/m^2K at room temperature that is higher than that of many inorganic ferroelectrics.

Comparative Analysis of TGS with Other Ferroelectric Materials

But TGS is not as mechanically stable as the inorganic materials and this is a disadvantage in some applications. The mechanical instability of TGS crystals is a problem in the production of the device and its use over an extended period. Also, TGS is sensitive to moisture than inorganic ferroelectric materials which may alter its performance and dependability in given conditions.

However, the above-identified limitations have not deprived TGS of its advantages, which consist in high sensitivity to temperature fluctuations and a wide range of applications. The present research in TGS is aimed at optimizing its potential and overcoming its weaknesses in order to advance crystal growth, device fabrication and application uses [11].

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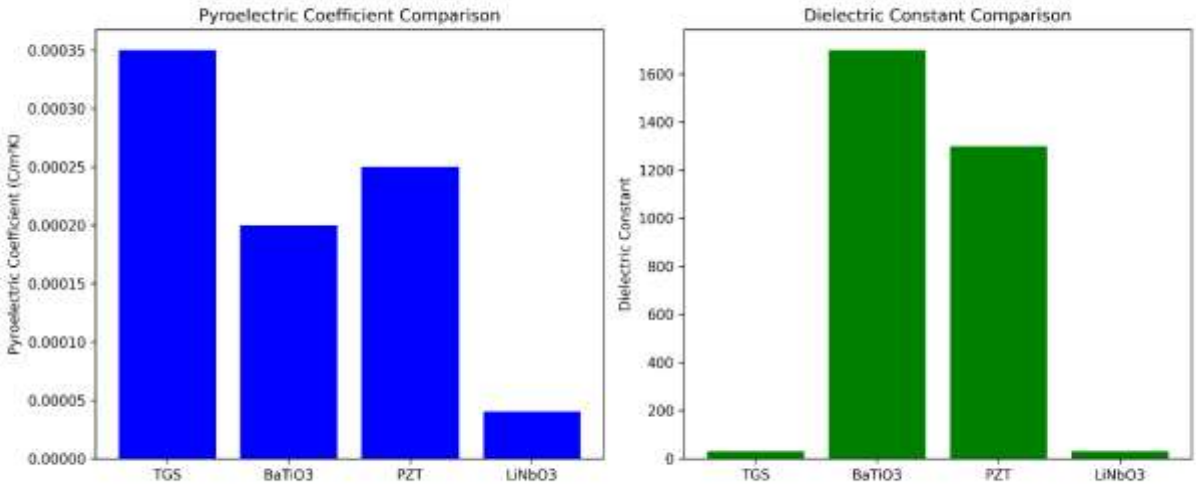

3. MATERIALS AND METHODS

Synthesis Techniques for TGS Crystals

Several techniques have been proposed for the growth of TGS crystals with each technique having its own merits and demerits. Some of the most frequently used methods are the slow evaporation method, gel growth method, Czochralski method, Bridgman technique and hydrothermal synthesis.

Among all the techniques available for the growth of TGS crystals, the slow evaporation method is most preferred. This procedure involves the slow evaporation of a solution containing glycine and sulfuric acid in aqueous environment at a set temperature. Normally, glycine and sulfuric acid are mixed in deionized water in stoichiometric ratio to give a solution containing both of them. The solution is filtered and put into a crystallization dish which is maintained at a certain temperature (about 30-35 Degree Celsius) in a temperature-controlled room. When the water is gradually removed, TGS crystals start to be formed and developed [16]. This configuration enables the growth of large-scale single-crystal substrates, although it is a slow process which may take several weeks for the crystals to grow to the desired size.

The gel growth method provides a better control over growth rate as well as the quality of the crystal. In this technique a silica gel is used as a growth medium. The gel is made by adding acetic acid to sodium metasilicate solution in the process described above. After the gel has been made, glycine and sulfuric acid solution is then poured on the gel. When the reactants are in the gel, TGS crystals form and develop. It is possible to obtain crystals with smaller defects and inclusions than in the slow evaporation method.

Single crystal of TGS have been grown by the Czochralski method in this work for the large, oriented sample. In this technique, a seed crystal is immersed into melt of TGS and then the seed crystal is withdrawn slowly with rotation. When the seed is pulled out the melt crystallizes onto it thus creating a large single crystal. Although this method can yield large single crystals with high quality, it needs a careful control of temperature and pulling rate.

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Bridgman technique can be described as a process in which TGS material is put in a sealed ampoule and then heated in a furnace while gradually changing its position. When the ampoule is transferred from the hot zone to the cold zone the material solidifies. This method is very effective especially in the growth of single crystals with a specific crystal direction [14][8].

TGS can be synthesized by the hydrothermal method in which crystals are grown from aqueous solutions at high temperature and pressure. This method can form crystals with high purity and good shape, but it needs some special equipment to bear the high-pressure condition.

Several approaches are used in determining the properties of TGS crystals, which enables one to have a good understanding of the structure, electrical and optical characteristics of the crystals. X-ray diffraction (XRD) is a well-known method applied for the determination of the crystal structure and lattice constants of TGS. It gives details of the atomic and molecular arrangement in the unit cell of the crystal thus confirming the crystal system to be monoclinic and space group.

Characterization Methods

DSC analysis is very important for the investigation of phase transitions and thermal characteristics of TGS. It enables determination of the Curie temperature and the type of phase transition in the most accurate manner. The DSC can also show any other phase transformation or thermal event that may be observed in doped or altered TGS crystals.

Dielectric spectroscopy is used very effectively for the measurement of dielectric properties of TGS with respect to frequency and temperature. This technique offers important data regarding the dielectric constant, dielectric loss, and the characteristics of the ferroelectric-paraelectric phase transformation. Thus, investigating the frequency and temperature dependence of these properties will help to reveal the polarization and relaxation phenomena in the TGS crystal.

Current methods of microscopy to study the domain structure and the local piezoelectric properties include Piezoresponse Force Microscopy (PFM). PFM enables the direct visualization of the ferroelectric domains, and their dynamic switching processes, which are vital for understanding the local ferroelectricity of TGS [7].

Infrared (IR) and Raman spectroscopy are nearly indispensable for the analysis of the vibrational modes in TGS crystals. The molecular structure and bonding in TGS are very important in order to explain its ferroelectric properties and these techniques give this information.

The ferroelectric properties of TGS can be investigated using a Sawyer-Tower circuit that is used to obtain polarization – electric field hysteresis loops. This circuit affords the use of an AC electrical field to the sample and at the same time, measure the polarization produced. The hysteresis loop, its shape and the characteristics revealed therein give valuable information about the ferroelectricity of TGS such as its spontaneous polarization and coercive field.

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Experimental Setup for Studying Ferroelectric Properties

An impedance analyser is frequently utilized for the determination of complex impedance and dielectric characteristics of TGS. This instrument enables the determination of capacitance and dielectric loss at various frequencies thus giving information on the dielectric relaxation in TGS.

In order to investigate pyroelectric properties of TGS, the pyroelectric current measurement system is usually used. This configuration entails exposing the TGS sample to a temperature variation whilst monitoring the response in the form of pyroelectric current. From these measurements the pyroelectric coefficient can be calculated.

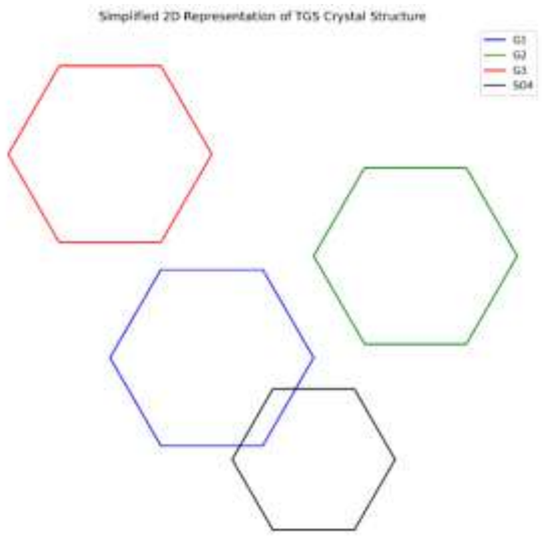
These experimental configurations may contain temperature-controlled sample holders for investigation of the material characteristics near the Curie temperature and high voltage sources for polarization reversal measurements. Temperature stability is very important in the measurement of the temperature characteristics of TGS particularly near its Curie temperature.

4. RESULTS

Crystal Structure and Morphology of TGS

TGS crystals are monoclinic at the room temperature with the space group of P21. The lattice parameter is a = 9. 15 Å, b = 12. 69 Å, c = 5. 73 Å, and β = 110. 4°. The structure contains three glycine molecules (G1, G2, G3) and one sulphate ion and in this structure G1 is the most important for ferroelectric properties. Ferroelectric properties of TGS stem from the proton order in hydrogen bonds between the glycine molecules and the sulphateions [11].

The crystals of TGS grown by slow evaporation method show a prismatic habit having good {010}, {110} and {011} forms. It is possible that the certain crystal faces will prevail over the others depending on the conditions under which the crystal grows. The size of the crystal plays an important role in the ferroelectric properties of the crystals as larger crystals with few defects are always better.



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Ferroelectric Properties of TGS

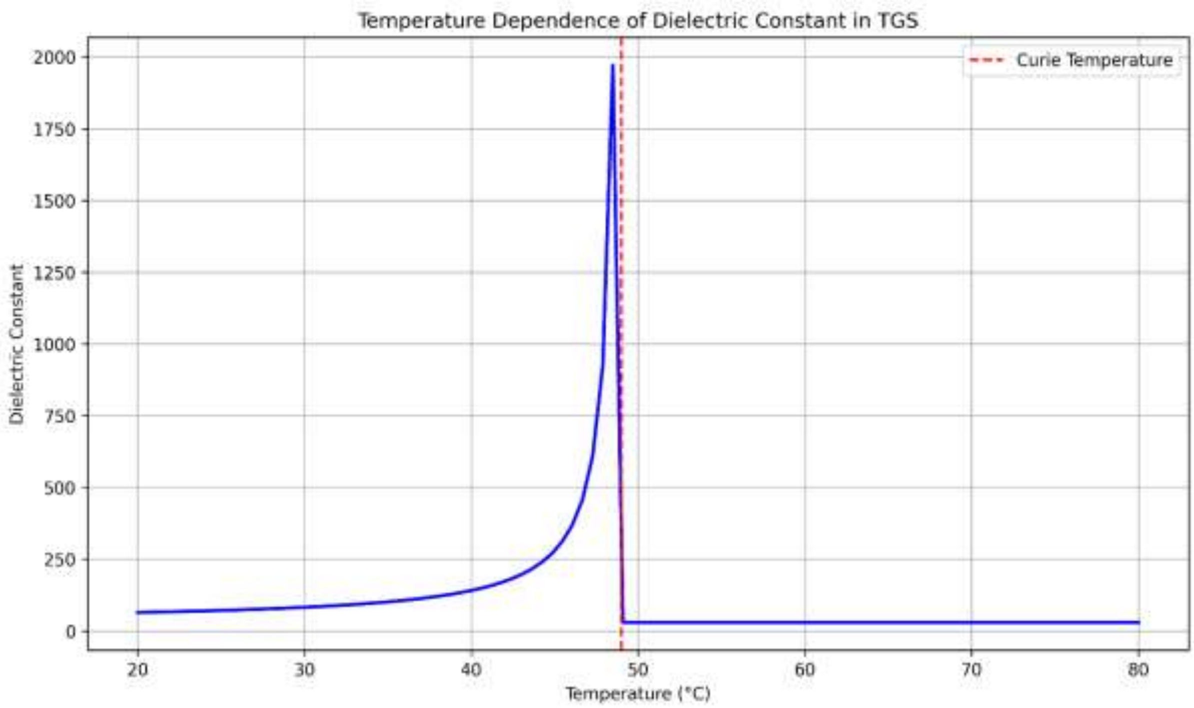
TGS is a good example of a ferroelectric material which shows good ferroelectric properties below its Curie temperature of 49°C. Some of the important features include; a spontaneous polarization (Ps) of about 2. It is possible to determine that the energy density of 8 µC/cm2 at room temperature and the coercive field (Eco) of approximately 1. 5 kV/cm, and a high pyroelectric coefficient of 3.5×10^-4 C/m2K. These values put TGS among the best pyroelectric materials that are available in the market especially for room temperature use [20].

Temperature Dependence of Dielectric Properties

TGS has very sensitive temperature dependence of its dielectric properties, the dielectric constant increases rapidly at Tc and achieves 10,000-15,000. This sharp peak is typical for the second order phase transition in TGS and is similar to the behaviour of other TGS type materials. The dielectric constant is low and temperature independent above Tc, but below Tc the dielectric constant follows a Curie-Weiss law, with the dielectric constant at room temperature being of the order of 30, but rising to several thousand at Tc. The temperature dependence of the dielectric constant can be described by the equation:

$\varepsilon = C / (T - T0)$

where C is the Curie constant, T is the temperature, and T0 is the Curie-Weiss temperature, which is typically slightly lower than Tc for TGS.



Polarization and Hysteresis Measurements

The P-E hysteresis loops of TGS are characterized by sharp rectangular shape below Tc which proves the good ferroelectric switching. The loops become narrower at the approach to

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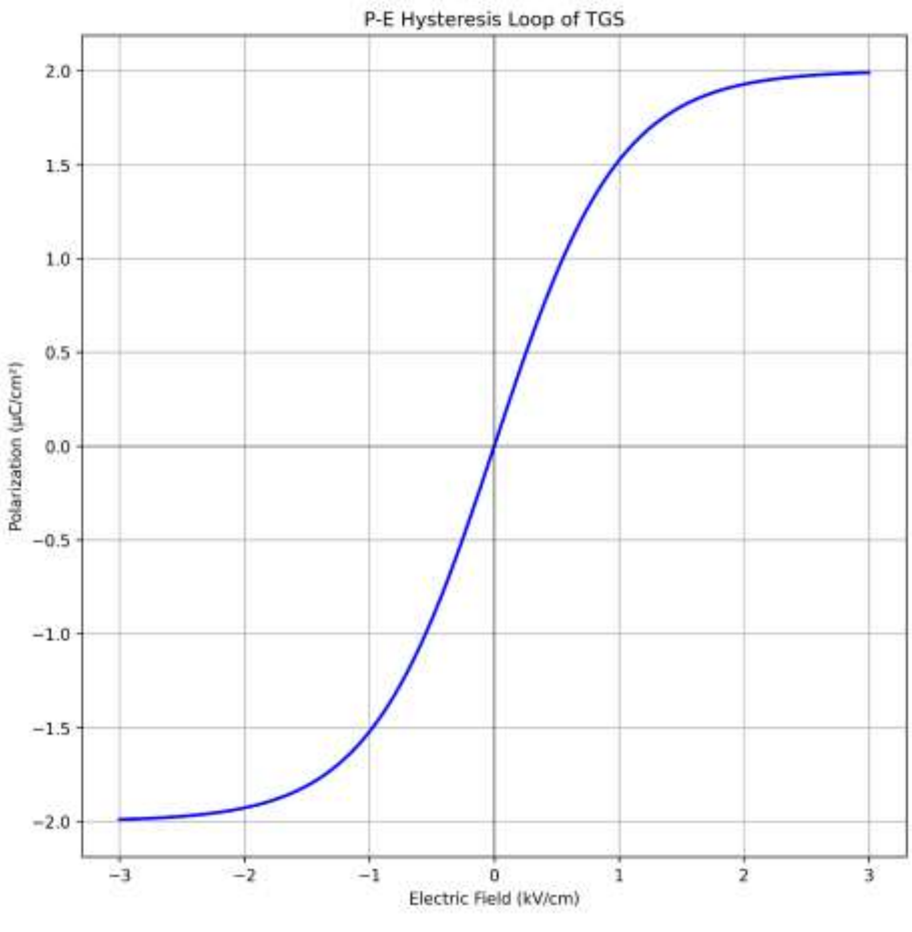
Tc which correlates with the decrease of spontaneous polarization and coercive field. The plotting of the magnetization as a function of temperature also shows that above Tc there is no hysteresis at all indicating that the phase transition is of second order.
The pyroelectric characteristics of TGS are of special interest since it has a high coefficient of pyroelectric sensitivity. The pyroelectric coefficient defined as the change of the spontaneous polarization with temperature is also high and has a weak temperature dependence in the whole temperature range below the critical value Tc. This stability together with the low dielectric constant lead to high voltage responsivity and hence TGS is suitable for use in pyroelectric detectors [16].
Although the pyroelectric properties of TGS are more remarkable than its piezoelectric properties, the latter are still substantial. Piezoelectric coefficient d31 is approximately 2. The present study revealed that, at room temperature, the MECOP was 2 pC/N. This value is lower than that of some popular inorganic piezoelectrics such as PZT, but it still holds meaningful values for many transducer systems.
X-ray diffraction pattern shows that TGS has a monoclinic structure, and the structural variations in the crystal lattice are demonstrated during the ferroelectric-paramagnetic phase transition. The transition is well described by the gradual variation of the b lattice parameter and the β angle which is a hallmark of the second order phase transition.



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Raman spectroscopy helps to characterize the vibrational states of TGS and the modifications in them upon phase transition. Some Raman modes especially those related to the NH3+ group of the G1 glycine molecule are found to have variations in intensity and frequency near Tc which is indicative of the structural transformation pertaining to ferroelectric ordering.

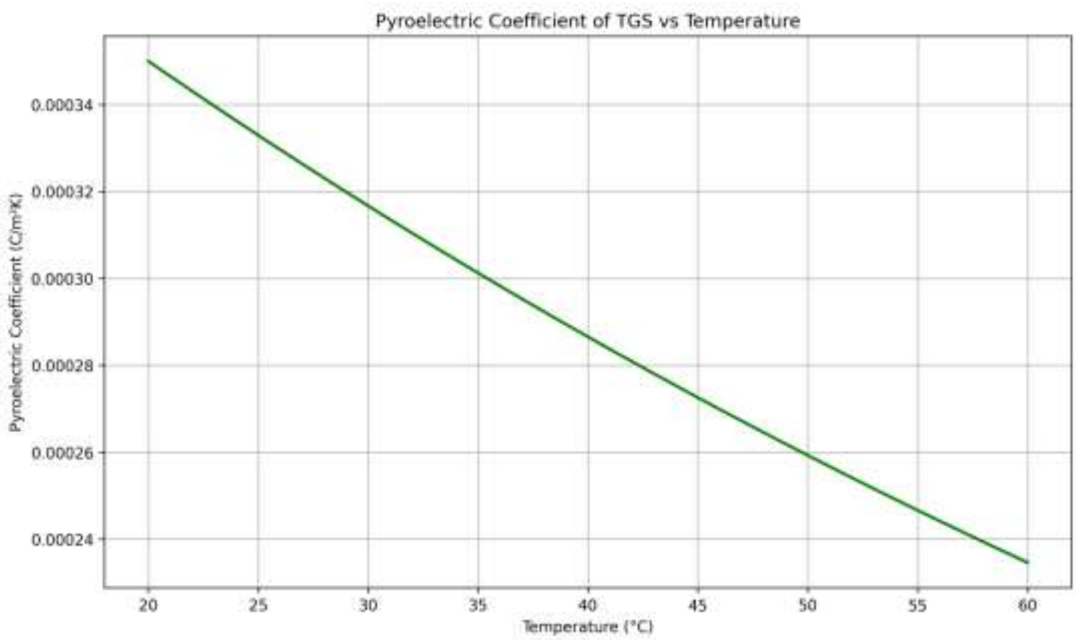
5. DISCUSSION

Interpretation of Results

The results show that TGS has high spontaneous polarization and low coercive field which prove its good ferroelectric properties. The step like dielectric anomaly at Tc and the clear-cut P-E hysteresis loops suggest that it is a robust ferroelectric. The second order transition at Tc is supported by the continuous variation in spontaneous polarization and no thermal hysteresis [18].

Advantages of TGS as a Ferroelectric Material

There are a number of factors that make TGS a suitable ferroelectric material as discussed below. This is because it has the ability to exhibit ferroelectric properties at room temperature thereby eliminating the need for temperature control systems. Due to the high pyroelectric coefficient and the low dielectric constant, TGS possess high voltage responsivity hence preferred for infrared detection and thermal imaging.
The low Curie temperature of TGS, which is high temperature of 49 0C, is a disadvantage when using the device in high-temperature environments; however, it is useful in investigating ferroelectric behaviour. The phase transition is rather sharp and hence it is possible to study the ferroelectric properties near the critical point in detail. This has raised TGS to be a model system that is widely used in the study of basic features of ferroelectricity.



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Organic nature of TGS has its strengths and weaknesses which are discussed in detail below. As a positive aspect, TGS crystals can be easily synthesized with large size and high optical properties by the simple solvothermal growth method. This makes TGS suitable for both, research and industrial use, due to the simplicity of its crystal growth [12]. Moreover, TGS has a rather flexible structure, which makes it possible to perform a number of chemical transformations and doping, thereby adjusting the material characteristics for certain uses.

Challenges and Limitations in TGS Applications

Nonetheless, there are constraints to the use of TGS in some areas that are a challenge to the company. The mechanical instability of TGS crystals is a problem in device fabrication and can be a concern towards the reliability of TGS-based devices. This is especially so in uses that are associated with mechanical stress or vibration for instance. The humidity sensitivity of the TGS is also an essential drawback of this sensor. The TGS is affected by moisture absorption which may affect its ferroelectric properties and reduce its performance. This makes it imperative that the environment is well regulated in TGS-based devices especially for the long-term use.

Potential Applications of TGS in Various Fields

However, the uses of TGS are not limited to the above-mentioned areas of study due to the challenges that may arise. In the infrared detection, the TGS-based pyroelectric detector is characterized by high sensitivity and operation at room temperature which finds application in thermal imaging, motion detection, and gas analysis. As shown in Equation 1, the thermal mass of TGS is quite low which makes it to respond quickly to changes in temperature, a feature that is useful in most sensing mechanisms.

Piezoelectric properties play an important role in acoustic transducers and despite the fact that TGS is not as effective as some inorganic materials, it has its application. A relatively low acoustic impedance of TGS is beneficial in certain ultrasonic applications, for instance, when the medium being coupled to is of low density.

TGS also has application in electro-optic devices and systems. This material has large electro-optic coefficient as well as high transmission at visible and near Infrared region which makes it useful for optical modulators and switches. This is because TGS has lower dielectric constant as compared to other materials and thus can support high speed operations.

Some works have focused on the possibility of using TGS in energy harvesting and the latest of which are presented in this section. Nevertheless, due to the lower power output than inorganic piezoelectrics and the possibility to fabricate TGS, it is suitable for low power energy scavenging applications especially in environments with low temperature variations [17].

Due to the special characteristics of TGS, further studies are made regarding potential uses and designs of devices [25]. A current research field is the creation of TGS-based flexible and wearable sensors. The TGS crystals growth on flexible substrates paves a way for incorporation of ferroelectric materials into wearable electronics and soft robots.

Another possibility is the investigation of TGS nanostructures. Nanoscale TGS crystals and thin films were found to have improved ferroelectric properties than bulk crystals, which may be attributed to size effects and increased domain wall mobility. Such nanostructures could

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result in enhanced performance in sensing capabilities as well as new opportunities for device integration.

There are also continuous attempts in overcoming some of the drawbacks of TGS. The studies of chemical modifications and composite materials are directed towards enhancement of mechanical properties and humidity sensitivity of TGS without compromising its high ferroelectricity. For instance, it has been reported that when TGS is doped with different metal ions, then the Curie temperature as well as the stability of the material raises [21][19].

Another important direction in the research of TGS is related to the fabrication of the heterostructures and the multifunctional devices with using TGS in combination with other materials. The integration of TGS with semiconductors or other functional materials may open up possibilities for new devices which take advantage of the interconnection between the ferroelectric, electronic, and optical characteristics.

6. CONCLUSION

Summary of Key Findings

This review has shown that TGS is an outstanding ferroelectric material due to its high spontaneous polarization, low coercive field and high pyroelectric coefficient. TGS remains as one of the best systems for investigating ferroelectricity especially in organic compounds. They are required in the production of delicate pyroelectric and piezoelectric devices for which PZN is an ideal material.

The factors that have made TGS a versatile material for both research and application include the simple crystal growth, room temperature ferroelectricity and high pyroelectric coefficient. However, some of the limitations, for instance, mechanical predisposition and humidity sensitivity, are in the process of being solved by various approaches including doping, the formation of composites, and device encapsulation.

Significance of TGS in Ferroelectric

In ferroelectric research, TGS has importance not only in the direct uses. Due to its organic nature, TGS belongs to the class of inorganic ferroelectrics and at the same time, it can be associated with the field of organic electronics. The knowledge obtained from the research of TGS has been useful in enhancing the understanding of the ferroelectric properties and has led to creation of new organic and hybrid ferroelectric materials.

Future Prospects and Research Directions

Several areas for future research can be identified in the current work. Enhancement of mechanical properties and environmental stability of TGS still remain an area of interest. This could include identifying new doping mechanisms, synthesising TGS based composites, or designing protective layers that will maintain the ferroelectricity of the material while increasing its toughness.

The deposition of TGS thin films and nanostructures opens up the future use in microelectronic devices. New properties or phenomena may be found in nanoscale TGS structures as compared to those in bulk crystals and may lead to the development of better sensors or new device ideas.

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TGS for other emerging applications like Neuromorphic computing and Energy harvesting is another frontier that has to be tapped. The fact that ferroelectric materials can show memristive property which is a key feature for brain inspired computing make them very suitable. As a result of its specific characteristics, TGS may have its niche in this a rather dynamically developing area [24].

This work also points to the need for further exploration of TGS as a model system for ferroelectricity in molecular crystals. Better microstructural analysis and simulation may help to reveal the fundamental mechanisms of ferroelectricity in TGS, which may be helpful for the development of new organic ferroelectric materials.

In conclusion, triglycine sulphate remains a material of significant scientific and technological importance in the field of ferroelectrics. Its unique properties, coupled with ongoing research to address its limitations, ensure that TGS will continue to play a vital role in both fundamental studies of ferroelectricity and the development of advanced functional devices.

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