

Article

Local Structural Distortions and Frustrated Magnetism in Slag-Derived Lithium Borate Glasses

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Abstract

This study analyzes the structural and magnetic properties of a new composite material with a lithium borate glass matrix loaded with electric arc furnace slag, with a composition of 50Li₂O-50B₂O₃-80 wt.% slag. The amorphous nature of the obtained glass is confirmed by X-ray diffraction analysis (XRD). Differential scanning calorimetry (DSC) indicates that the material has an extremely high thermal stability as evidenced by its high glass transition temperature which is due to the reinforcing role of the slag metallic oxides. Additionally, magnetic studies reveal a highly frustrated magnetic state in the disordered system. The significant addition of mixed-valence iron ions (Fe²⁺/Fe³⁺) in the slag forms antiferromagnetic interactions in the system. These factors result in the stabilization of the spin-glass-like state. The incorporation of slag within the lithium borate matrix is a promising route to valorize this industrial by-product and to develop new functional materials.

Keywords: electric arc furnace slag; borate glass; X-ray diffraction; DSC; antiferromagnetic interactions



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1. Introduction

Industrial slag is considered one of the most significant types of solid wastes globally. The majority of it is generated during the production of metals and electricity. Extraction of such waste is vital not only due to environmental considerations but also from an economic perspective. Currently, the main approach to recycling is to reuse slags as substitutes of natural aggregates in various structures in order to reduce CO₂ emissions and increase resource utilization efficiency [1–3]. Electric arc furnace slag (EAFS) results from the smelting process of liquid steel. It mainly contains oxides of calcium, iron, and silicon that hardly react with each other. Therefore, EAFS is suitable as a replacement material for natural aggregates in Portland cement-based materials [4,5]. Research indicates that EAFS can be used as a replacement of natural fine aggregates in ultra-high-performance

concrete (UHPC) without decreasing its properties [6,7]. The fact that EAFS has an oxide-rich chemistry, especially its high iron content, suggests another possibility that has barely been explored: using it as a raw material for advanced functional glasses.

Borate glasses have attracted attention due to their optical transparency, low melting points, thermal stability, and ability to dissolve rare-earth and transition-metal ions [8,9]. The structure of pure B_2O_3 glass is made up of trigonal boroxol rings (BO_3) and tetrahedral four-fold coordinated boron (BO_4). Unlike silicate glasses that have rigid tetrahedral networks, the borate framework is topologically flexible. This flexibility allows the network to be modified by adding network modifiers or intermediates [10,11]. With its ability to dynamically reorganize its building blocks, B_2O_3 is an ideal base matrix for the design of customized amorphous materials for applications such as solid-state electrolytes, non-linear optical devices and radiation shielding [12–14].

Lithium borate glasses exemplify this flexibility and are governed by the well-known “borate anomaly” [15]. Pure B_2O_3 glass contains only trigonal [BO_3] units in boroxol rings. When alkali oxides such as Li_2O are added, the trigonal units [BO_3] convert to tetrahedral [BO_4] groups, creating bridging oxygens (BOs) that improve thermal stability, humidity resistance, and glass-forming ability [16]. However, past a certain modifier threshold, the coordination reverses: [BO_4] converts back to [BO_3], producing non-bridging oxygens (NBOs) [17,18]. The 50 Li_2O -50 B_2O_3 composition is a useful reference point in this respect. Its amorphous framework is well-defined and responsive enough to serve as a baseline for structure–property studies in glass science [19].

This binary borate network can also be modified with transition-metal (TM) oxides. This has led to research on solid-state battery cathodes, solar energy conversion, electronic switching circuits, and electrochemical gas sensors [20,21]. In the glassy matrix, TM ions typically exist in mixed-valence states, altering their local coordination environment and enhancing the non-linear optical, electronic, and magnetic properties of the host [22,23]. Most published work has involved adding pure synthetic oxides individually to lithium borate glasses to adjust their properties. This approach relies on expensive, refined chemical precursors. Our approach is different in that we replace those refined oxides with a single complex industrial by-product: EAFS. Adding 80 parts of slag to the glass dramatically changes the simple binary baseline, introducing several network formers and modifiers at once. Instead of merely encapsulating waste or using it as a low-value aggregate, this work treats the slag as a functional material precursor.

In order to examine the properties of this glass, it is necessary to understand the structural interactions of major oxides present in the slag. In the case of Fe_2O_3 , magnetic moments are formed, which contribute to making the structure magnetic. These are multi-valent cations that occupy network forming and modifying sites, and due to their proximity in the tightly packed borate network, there are strong anti-parallel super-exchange Fe-O-Fe interactions [24–26]. Iron may be present as Fe^{2+} and Fe^{3+} and the relative proportion of these ions depends on such factors as melting temperature, glass composition and iron content. Fe^{2+} ions ($3d^6$ configuration) normally occupy octahedral coordination sites and show a spin-allowed electronic transition. In contrast, Fe^{3+} ions ($3d^5$) normally do not show spin-allowed transitions, thus influencing the spectroscopic behavior of the glass [27,28]. Calcium oxide (CaO) further modifies the network, in which Ca^{2+} cations occupy interstitial sites. This compensates for the negative tetrahedral units’ charge and generates non-bridging oxygens (NBOs) which affect packing density and mechanical stability [29,30]. Silica (SiO_2) transforms the matrix into a lithium borosilicate network by polymerizing the framework through B-O-Si linkages which improves chemical and thermal durability [31,32]. With 80 parts slag, these oxides are not isolated dopants. Their combined effect determines the distribution, spacing, and coordination environments of

iron ions. Therefore, the magnetic properties, such as susceptibility and low-temperature exchange interactions, are a direct consequence of this locally engineered borate network.

However, it is also fascinating to observe that the properties of condensed matter systems are defined by the notion of symmetry. The crystal structures, where the arrangement of atoms takes on a long-range periodicity, impose certain symmetry operations. In contrast, amorphous materials lack translational symmetry over long distances but preserve a certain degree of local structural order through short-range coordination units [33,34]. The local symmetry around network-forming and network-modifying species strongly influences bond lengths, bond angles, and electronic environments, thereby controlling the emergence of collective physical phenomena [35,36]. Consequently, understanding how local symmetry evolves in complex glassy networks is essential for establishing structure–property relationships.

From a magnetic perspective, symmetry breaking has profound consequences on exchange interactions between magnetic ions. The type and extent of super-exchange routes depend highly on the presence of local coordination polyhedron distortion, affecting both bond geometry and interatomic distances. In disordered systems containing transition-metal ions, the coexistence of multiple local environments generates a broad distribution of exchange interactions, often leading to competing ferromagnetic and antiferromagnetic couplings [37,38]. This competition creates magnetic frustration, suppresses long-range magnetic ordering, and may stabilize unconventional magnetic states such as spin-glass phases [39,40]. Thus, it could be said that the decrease in local structural symmetry is one of the main causes behind the complex magnetic properties found in many amorphous compounds.

Glassy systems, characterized by the combination of variable chemical–structural disorders, provide a perfect ground for investigating the interaction between symmetry and magnetism. Glasses provide a continuous variety of local environments that can be designed by adding network modifiers and transition-metal species, in contrast to crystalline materials where symmetry is limited by periodicity. Such research into the effects of symmetry breaking in local regions on the magnetic interaction at the atomic level is now possible thanks to this flexibility. The distribution of $\text{Fe}^{2+}/\text{Fe}^{3+}$ ions in an inherently disordered structure in iron-based borate glasses offers an ideal medium to explore the relationship between spin glassiness and magnetic frustration.

In this context, the present study proposes a novel, high-value approach to valorization by designing a multi-oxide glass that is heavily loaded with industrial waste. Specifically, a new glass system with the composition $50\text{Li}_2\text{O}-50\text{B}_2\text{O}_3-80\text{ wt.\% slag (LiBS)}$ was synthesized. This study primarily aims to elucidate the direct correlation between the structural compactness induced by this extreme waste loading and the macroscopic magnetic properties of the resulting material. The amorphous network topology and local environment were thoroughly characterized using structural–physical measurements and vibrational spectroscopy (FTIR and Raman). Concurrently, we conducted a detailed investigation of the magnetic properties using vibrating sample magnetometry (VSM) as a function of magnetic field and temperature. These findings reveal that confinement of iron ions in the lithium borate glass matrix leads to strong antiferromagnetic behavior. This opens new perspectives for developing functional magnetic materials from the circular economy.

2. Materials and Methods

This research used electric arc furnace slag (EAFS) as a natural raw material for the production process. The EAFS material was collected at a mining location situated in the Oujda region of Morocco. Before the glass manufacturing process, the slag material was crushed and powdered in a laboratory-grade agate mortar to facilitate homogeneous

mixture and reduce potential analytical interferences. The EAFS was characterized in terms of its chemical composition by Panalytical XRF (PW2400, PANalytical B.V., Almelo, The Netherlands).

The glass samples with the composition of 50Li₂O-50B₂O₃-80 wt.% slag (LiBS) were prepared using a traditional melt-quenching method, utilizing high-purity analytical-grade raw materials such as lithium carbonate (Li₂CO₃) and boric acid (H₃BO₃) and electric arc furnace slag. Specifically, the base glass matrix with a stoichiometric composition of 50Li₂O-50B₂O₃ (expressed in mol%, representing a 1:1 molar ratio of lithium oxide to boron trioxide) was prepared. Electric arc furnace slag (EAFS) was incorporated into this precursor mixture as an overweight addition of 80 wt.% (i.e., 80 g of EAFS was added per 100 g of the base glass matrix).

The appropriate amounts of the various starting materials were blended in an agate mortar and thoroughly homogenized prior to transfer to a platinum crucible and heated in an Lenton electrical muffle furnace (Lenton, Hope Valley, UK) at 200 °C during 12 h. Then they were heated at 600 °C for 24 h to ensure the elimination of the volatile H₂O, NH₃, and CO₂ compounds. The temperature of the muffle was then raised to 1000 °C and maintained at this level for 2 h to allow for the complete smelting and refining of the resulting liquid. The melt was then rapidly quenched by placing the crucible on a stainless-steel plate. The resulting glass samples were then placed in a desiccator to prevent any possibility of moisture absorption prior to structural and magnetic property characterization.

The surface morphology and macroscopic homogeneity of the synthesized glass samples were initially examined using an Olympus BX51M (MM03-01, Olympus Corporation, Tokyo, Japan) optical microscope in reflected light mode. Concurrently, a Quanta 200 FEG scanning electron microscope (SEM; FEI/Thermo Fisher Scientific, Waltham, MA, USA) was utilized to investigate the microstructural features and cross-sectional fracture topologies at the sub-micron scale, further verifying the structural uniformity of the amorphous framework.

The amorphous nature was checked using a Siemens D5000 diffractometer (Siemens AG, Karlsruhe, Germany) with CuK α radiation over a 2 θ range of 5° to 90°. Thermal properties were analyzed by differential scanning calorimetry (DSC) with a Setaram121 calorimeter (Setaram Instrumentation, Caluire-et-Cuire, France) at a heating rate of 10 °C/min under Argon flux. Densities were determined using a RADWAG analytical balance (RADWAG Balances and Scales, Radom, Poland) and the Archimedes technique with water as the immersion liquid. Structural features of the glass samples were characterized by Fourier-Transform Infrared Spectroscopy (FTIR) and Raman spectroscopy. FTIR analysis was conducted with Bruker Platinum-ATR apparatus (Bruker Corporation, Billerica, MA, USA) in the 400–1800 cm^{−1} range. Raman spectra were collected using a Perkin-Elmer Spectrum GX spectrometer (PerkinElmer, Inc., Waltham, MA, USA) with a 532 nm excitation laser, recorded in backscattering geometry from 200 to 1800 cm^{−1}.

The magnetic properties were characterized using a Physical Property Measurement System (PPMS DynaCool, Quantum Design, San Diego, CA, USA) under various applied external fields. Magnetization (M(T)) was measured as a function of temperature from 15 to 300 K, with applied magnetic fields ranging from 0.1 to 10 kOe, in both zero-field-cooled (ZFC) and field-cooled (FC) regimes.

3. Results and Discussion

3.1. X-Ray Fluorescence

The elemental composition of electric arc furnace slag (EAFS), which was used as the raw material to make the glass materials, can be found in Table 1. The results show that the main chemical elements in EAFS are Fe₂O₃ (31.69 wt%), CaO (30.59 wt%) and

SiO₂ (14.21 wt%). These three components are more than 76% of the total mass of the slag. Additionally, minor constituents such as Al₂O₃ (8.30 wt%), MgO (6.16 wt%) and MnO (3.61 wt%) complement the network modification by altering local field asymmetries, while the negligible amounts of impurities (SO₃, K₂O, TiO₂ and P₂O₅) confirm that the selected EAFS possesses an ideal chemical readiness for high-value upcycling into functional glass materials.

Table 1. Chemical composition of electric arc furnace slag.

Constituent	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	SO ₃	K ₂ O	TiO ₂	MnO	MgO	P ₂ O ₅
Content %	14.21	8.30	31.69	30.59	0.46	0.02	0.59	3.61	6.16	0.57

3.2. Morphology and Microstructure

Figure 1 shows the morphology and optical microstructure of the studied sample. In Figure 1a, the sample appears as a transparent to translucent amber-yellow glass. The successful formation of a continuous glassy phase is deduced from the good visual homogeneity and by the absence of visible cracks or significant opacity, despite the high slag content.

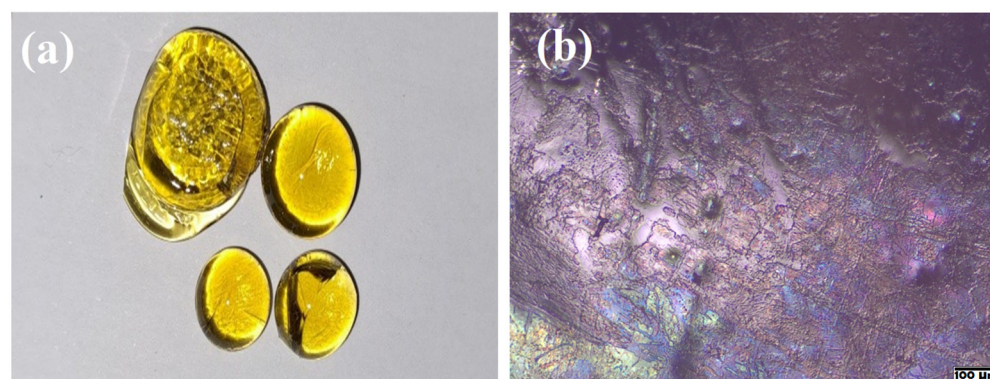


Figure 1. (a) Photograph of the as-prepared LiBS sample, exhibiting a transparent amber-yellow appearance. (b) Optical microscopy image of the sample.

The optical microscopy image (Figure 1b) shows a mostly homogeneous glass matrix with slight local contrast variations and a few brighter regions. These heterogeneities may be associated with slag-enriched microdomains, but could be also associated with the minor glassy compositional fluctuations. It is worth mentioning that neither large crystallized regions nor well-defined crystalline grains are clearly observed, highlighting the amorphous nature of the studied sample. Furthermore, the multi-color contrast observed in the micrograph could be linked to the interference phenomenon and/or to diffraction of the light. These preliminary observations tend to confirm that the slag was well incorporated into the lithium borate matrix, thus leading to a homogeneous glass; however, a small tendency toward phase separation or crystallization could be spotted.

Figure 2 exhibits SEM images of the glassy system LiBS. As depicted in the figure, compact and homogeneous morphology seems predominant, a signature of an amorphous material. In addition, the fracture surface shows a limited number of pores with an irregular glassy region containing dispersed bright particles.

It should be noted that despite the high slag content, no extensive grain boundaries are observed, confirming the formation of a vitreous matrix. However, at higher magnifications, some small particles embedded within the glass network are detected and may originate from slag-derived phases. The obtained homogeneous distribution and the

non-observation of any significant interfacial separation reveal the good incorporation of the slag constituents within the glassy matrix.

To further assess the chemical homogeneity of the glass network, EDS elemental mapping was performed on the sample (Figure 2b). The spatial mapping of all detected constituents including Li, B, O, Fe, Mg, Al, Si, and Ca demonstrates a highly uniform elemental distribution across the entire scanned area. The absence of localized clusters or concentration gradients for any of these elements, particularly for the heavy metal components (Fe, Si, Ca), confirms that the EAFS has been completely and homogeneously incorporated into the amorphous borate matrix during the melt-quenching process.

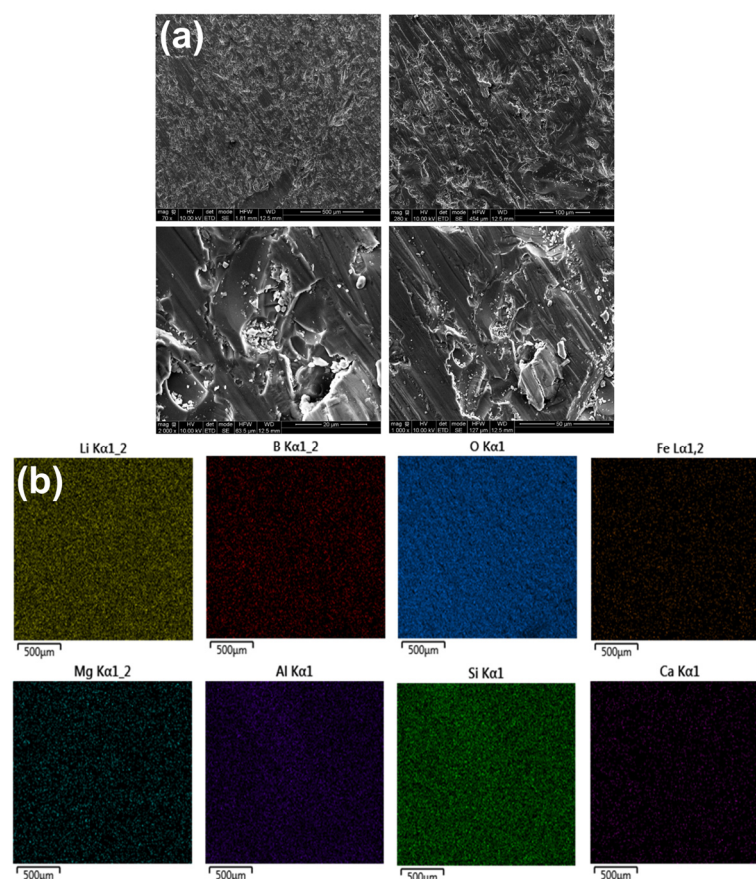


Figure 2. (a) SEM micrographs of the 50Li₂O-50B₂O₃-80 wt.% slag (LiBS) glass recorded at different magnifications. (b) EDS elemental mapping of the major constituents within the glass network.

3.3. Structural and Thermal Properties

The non-crystalline and amorphous characteristics of the prepared LiBS glass can be seen in Figure 3a. The analysis of XRD shows broad, diffuse halos throughout the pattern, which reflects the amorphous and long-range disorder associated with the glassy network. This phenomenon is also important because it reflects the existence of a certain amount of disorder within glass materials as opposed to a much more regularly ordered array of atoms in crystalline solids.

The thermal stability and structural evolution of the synthesized glass matrix was evaluated by differential scanning calorimetry (DSC), as shown in Figure 3b. The thermogram displays a well-defined endothermic step corresponding to a glass transition temperature (*T_g*) of 465 °C, determined as the onset temperature using the tangent construction method shown in Figure 3c. The limited thermal window of this DSC analysis (100–700 °C) captures the glass transition but does not extend to melting temperatures (likely >1000 °C given the high refractory oxide content). Determination of the melting point and melting

mechanism (congruent vs incongruent) would require high-temperature DSC or complementary techniques and represents a direction for future investigation. The high T_g value is an indication of improved structural rigidity as compared to typical baseline borate networks and is strongly correlated to the profound structural modifications induced by the addition of the EAFS. Since calcium and iron oxides are the main components of the used slag, their simultaneous introduction into the matrix leads to significant changes in the cross-linking density of the network [41,42]. The incorporation of these stable metal oxides provides a strong structural reinforcement to the amorphous framework, increasing the overall connectivity and rigidity of the vitreous skeleton and thus conferring the glass with superior macroscopic thermal stability [43]. Besides the identification of the glass transition, the DSC profile yields important information on the long-range structural stability of the matrix against devitrification. In particular, the heat flow curve is remarkably flat in the broad temperature range 500 °C to 700 °C, and it exhibits a complete absence of exothermic crystallization peaks (T_c). The absence of devitrification features means that, despite the very high loading of multi-oxide slag, the network has excellent structural integrity in a large temperature window [44]. This thermal behavior is an important scientific guarantee for the further study of the magnetic properties of the material. It rigorously excludes the formation of any localized crystalline clusters or parasitic magnetic phases, which could otherwise generate extrinsic magnetic responses.

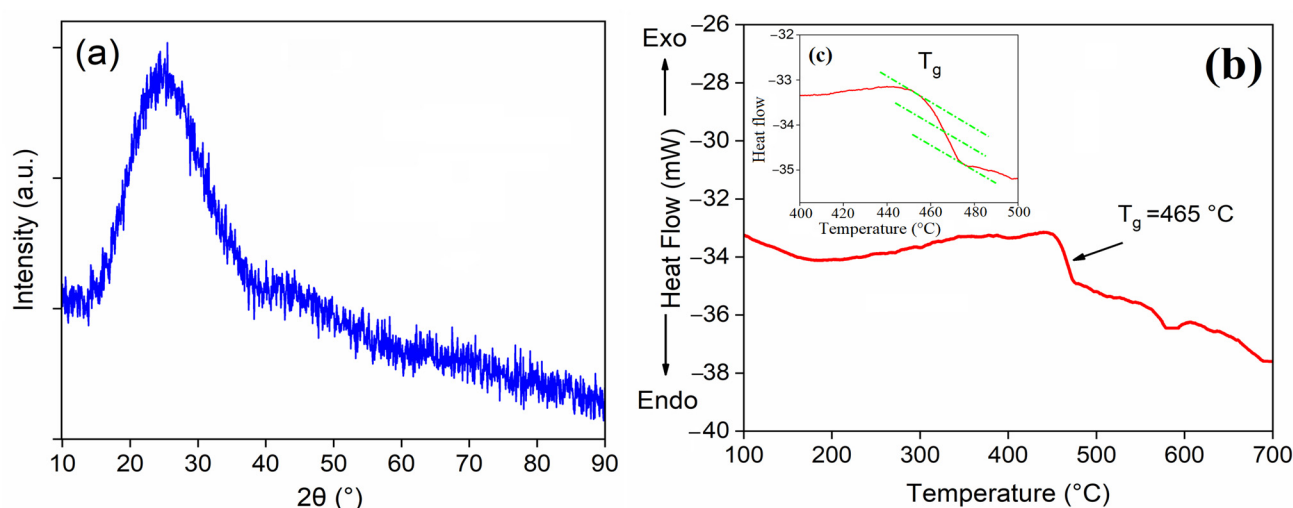


Figure 3. (a) XRD patterns, (b) DSC curve of the LiBS glass and (c) enlarged view of the glass transition region showing the green lines used for the tangent construction to determine T_g .

A minor endothermic deflection is observed near 600 °C. However, given its broad profile and minor enthalpy, this feature is attributed to an instrumental baseline drift, a common thermal artifact related to the experimental setup at higher temperatures, rather than a true structural or thermodynamic phase transition within the glass network. Therefore, it does not represent macroscopic crystallization or phase separation, confirming that the amorphous integrity of the sample is fully maintained up to 700 °C.

Finally, it is important to note that the DSC heating rate of 10 °C/min represents a standard characterization protocol for identifying thermal transitions in already-prepared glass samples and does not reflect the rapid quenching conditions (>100 °C/min) applied during original synthesis. The fact that the sample remains amorphous and stable during slow-heating DSC scans confirms that the original quenching successfully produced a true glass without unintended crystallization. No crystallization peaks (T_c) appear during the entire 100–700 °C heating range, providing strong evidence that the as-prepared material is indeed an amorphous solid, not partially crystallized.

This structural arrangement and the resulting network compaction are further substantiated by the physical property measurements, namely density and molar volume (V_M). The density of the glass was determined to be $2.33 \text{ g}\cdot\text{cm}^{-3}$, while the corresponding molar volume was calculated at $59.78 \text{ cm}^3\cdot\text{mol}^{-1}$. These physical values reflect a characteristic packing density that is primarily driven by the large-scale accumulation of heavy alkaline-earth metal ions (Ca^{2+}) and transition-metal ions ($\text{Fe}^{2+}/\text{Fe}^{3+}$) supplied abundantly by the 80 parts of slag [45,46]. In this type of glass, calcium and iron ions act as modifiers of the network. These ions fill interstitial areas located in the amorphous structure of the glass. They serve as charge neutralizers on the tetrahedral units $[\text{BO}_4]^-$ [47]. Since there is such a high concentration of calcium and iron in the structure of the glass, they are both tightly packed, making the overall structure of the glass dense and well-defined.

To further elucidate the local structural network and structural units of the synthesized LiBS glass, FTIR and Raman vibrational characterizations were investigated, as illustrated in Figure 4a,b. Both complementary vibrational techniques provide key insights into the short-range order of this multicomponent system, mapping the complex co-polymerization between the lithium-borate host and the high concentration of slag-derived oxides. The broadness of the vibrational bands observed in both spectra confirms the structurally disordered, fully amorphous nature of the sample, which perfectly aligns with the previous XRD and DSC observations. The FTIR transmittance spectrum is given in Figure 4a. The obtained absorption bands were compared to the equivalent materials reported in the literature. Table 2 gathers the position of bands and their related assignments. The band around 1370 cm^{-1} is linked to the asymmetric stretching vibration of the B-O bond in BO_3 units. It should be noted that this band has been previously identified in the orthoborate (BO_3^{3-}), metaborate (BO_2O^-) and pyroborate ($\text{B}_2\text{O}_2\text{O}_2^{2-}$) groups [48,49]. Meanwhile, the band at 1240 cm^{-1} is attributed to the stretching vibrations of terminal B-O bonds in $[\text{BO}_3]$ units, suggesting the presence of non-bridging oxygen (NBO) atoms which are heavily generated by the modifier effect of Ca^{2+} and iron ions [50,51]. The band at around 1045 cm^{-1} is due to the stretching vibrations of the B-O bond in BO_4 units from tetraborate, pentaborate and triborate units [52,53]. The coexistence of these bands clearly confirms that the slag components are not simply encapsulated but actively network-integrated. Those near the 920 cm^{-1} range are associated with stretching vibrations of B-O bonds in tetrahedral $[\text{BO}_4]$ units originating from di-borate structural groups [54,55]. Moreover, the bands observed around 703 cm^{-1} are attributed to Si-O bending [56,57], and to the bending vibrations of B-O-B linkage in the borate network [58,59].

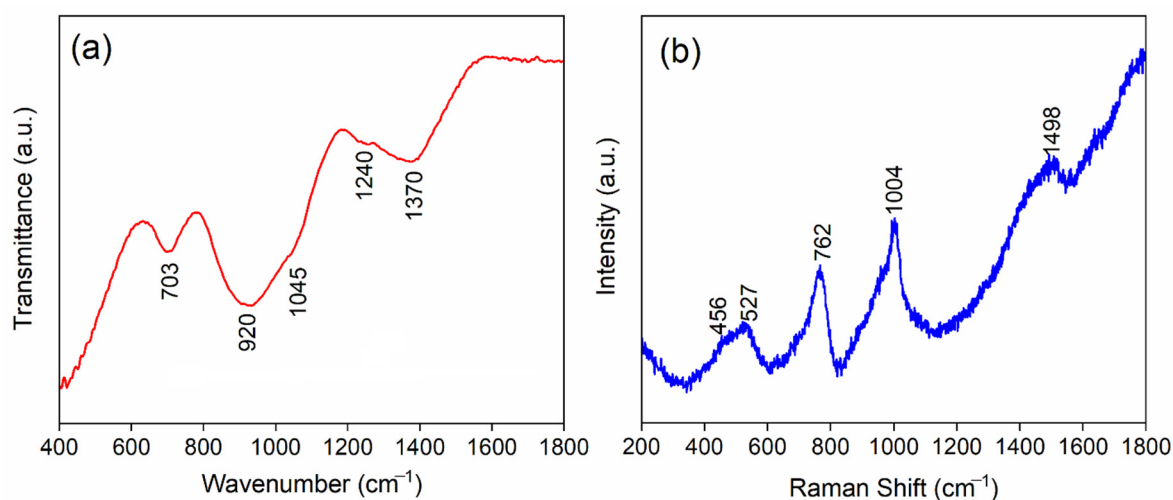


Figure 4. (a) FTIR (red line) and (b) Raman (blue line) spectra of LiBS glass.

Table 2. Infrared and Raman wavenumbers and their corresponding assignments for the LiBS sample.

FTIR	Raman	Assignments	References
1370	1498	Asymmetric stretching vibration of B-O bond in $[\text{BO}_3]$ units present in metaborate, pyroborate, and orthoborate groups	[48,49,60,61]
1240	-	Stretching vibrations of terminal B-O bonds in $[\text{BO}_3]$	[50,51]
1045	1004	Stretching vibrations of B-O bond in BO_4 units from tetraborate, pentaborate and triborate units	[52,53,62,63]
920	-	Stretching vibrations of B-O bonds in tetrahedral $[\text{BO}_4]$ units originating from di-borate structural groups	[54,55]
-	762	Six-membered rings containing one trigonal $[\text{BO}_3]$ and two tetrahedral $[\text{BO}_4]^-$ borate ring-breathing vibrations	[64,65]
703	-	Si-O bending Bending vibrations of B-O-B linkage in borate network	[56,57] [58,59]
-	527	Bending vibrations of BO_3^{3-} units	[49,66]
-	456	Bending vibrations of B-O-B linkages in the borate network	[67,68]

The Raman data was collected in the range $200\text{--}1800\text{ cm}^{-1}$, which is presented in Figure 4b. The summary of the vibrational band assignments is given in Table 2. The bands found at 1498 cm^{-1} are assigned to the asymmetric stretching vibrations of B-O bonds in BO_3 triangles [60,61]. The bands located at 1004 cm^{-1} can be attributed to the stretching modes of B-O bonds in BO_4 tetrahedra [62,63]. Meanwhile, the bands found at 762 cm^{-1} can be assigned to the ring-breathing mode of six-membered rings containing $[\text{BO}_3]$ and $[\text{BO}_4]^-$ units, providing direct spectroscopic proof of the borate anomaly stability even under an 80 wt.% of slag [64,65]. The band observed at 527 cm^{-1} corresponds to the bending vibrational mode of BO_3^{3-} units [49,66]. The band at 456 cm^{-1} is attributed to the bending vibrations of B-O-B linkages in the borate network [67,68].

The vibrational modes identified through the FTIR and Raman spectroscopy provide insights in understanding the short-range order and spatial symmetry development in the glass matrix. In an ideal glass structure, the local environment is composed of symmetrical trigonal and tetrahedral structures. Due to their geometry, they are presented in degenerated vibrational modes where there should be sharp spectral peaks. However, the introduction of the multi-oxide EAFS destabilizes the structural equilibrium of the glass structure. The influx of heavy oxide network modifiers and formers causes extensive spatial and chemical instability. As a result, the clear manifestation of the broadening, asymmetry, and overlap of the stretching bands of FTIR and Raman spectra indicate that the structures are far from being symmetric and ideal. The presence of such a huge amount of EAFS necessitates distortion in the structures, leading to varied distributions of bond lengths and broken bond angles in the vicinity of the central atom. In the context of vibrational spectroscopy, this localized symmetry breaking manifests directly as the lifting of vibrational degeneracy and overlapping of sub-bands, creating the broad spectral envelopes characteristic of the amorphous state. Ultimately, this reduction in the symmetry at the microstructure level is the core characteristic of the synthesized glass. This clearly shows

that the atoms are arranged in an asymmetric manner within the atomic field, and this directly forms the basis of its thermal and mechanical stability.

3.4. Magnetic Investigation

To evaluate the magnetic characteristics of the LiBS glass, M-H magnetic hysteresis loops were recorded at different temperatures ranging from 15 K to 300 K, as shown in Figure 5. It is evident that all samples displayed non-linear M-H loops at each of the selected temperatures, which do not reach saturation even under the application of the highest magnetic field (10 kOe). All samples demonstrate distinct antiferromagnetic (AFM) behavior based on the morphology of both hysteresis loops [69]. The antiferromagnetic coupling is a further result of the Fe-O-Fe exchange interaction. Between temperatures of 15 K and 50 K, the linear dependence of magnetization in high fields is accompanied by an increase in maximum magnetization. The maximum magnetization approaches 0.6 emu/g at about 15 K, suggesting a strong paramagnetic background from intermixed Fe^{2+} and Fe^{3+} ions within the amorphous borate matrix as well as possible short-range antiferromagnetic interactions. In contrast, these curves evolve into a well-defined S-shaped hysteretic form at a temperature of 300 K; indeed, the maximum magnetization is decreased by up to an order of magnitude (≈ 0.08 emu/g). The room-temperature antiferromagnetism is due to the antiferromagnetic super-exchange interactions between neighboring Fe ions distributed within the disordered glass network. The macro-spins of these clusters are free to flip under thermal energy but can easily be aligned in an external magnetic field [70,71].

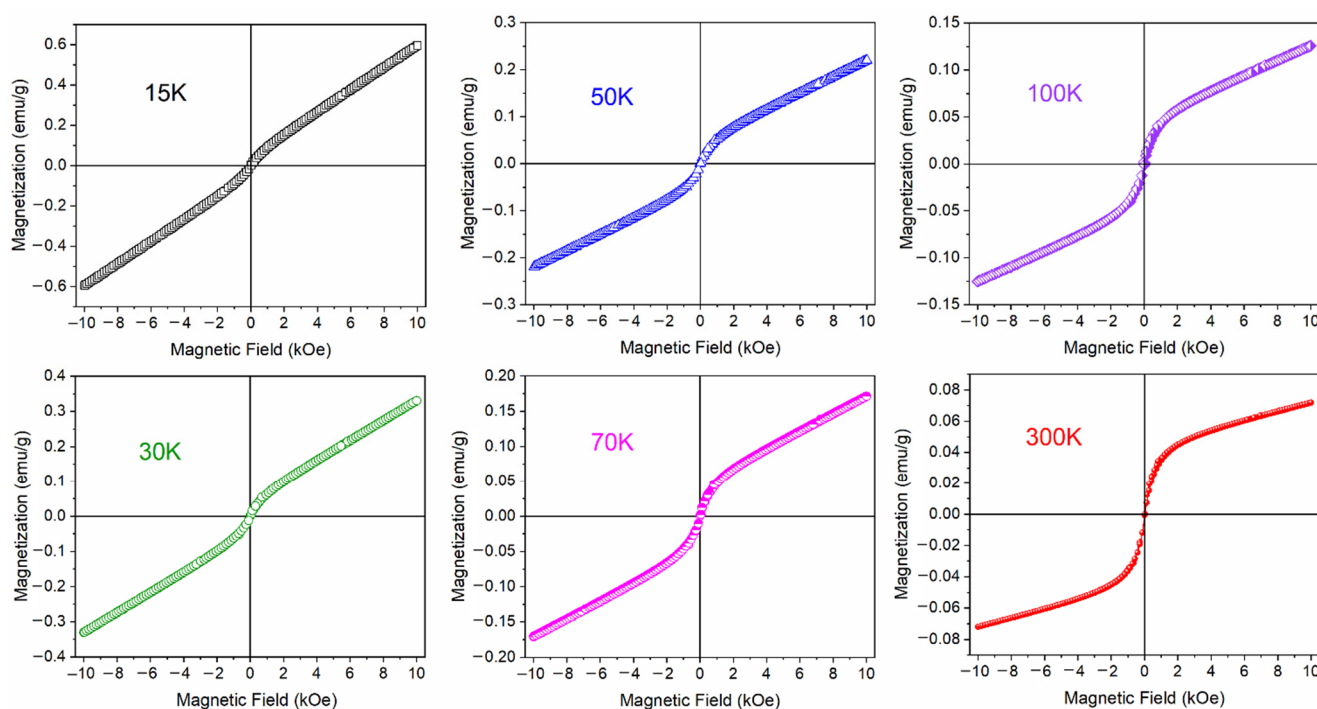


Figure 5. Magnetic hysteresis loops (M-H) of the LiBS glass recorded at various temperatures from 15 to 300 K.

To investigate the magnetic characteristics of the synthesized material, the zero-field-cooled (ZFC) and field-cooled (FC) magnetic susceptibility were measured under different magnetic fields, as shown in Figures 6 and 7. It is important to note that in the ZFC and FC graphs, a bifurcation occurs starting at $T = 275$ K when a magnetic field of 0.1 kOe is applied. This phenomenon is commonly associated with spin-glass-like behavior [72,73]. The observed bifurcation decreases as the magnetic field strength increases, ultimately converging above 10 kOe (see Figure 6). This dependence on the magnetic field is intrinsically linked

to the significant spin frustration that exists within the amorphous material; the application of a high external magnetic field effectively overcomes these local energy barriers, forcibly aligning the frustrated spins along the field direction even at low temperatures [74,75]. For a magnetic phase transition of a system, a strong splitting between ZFC and FC protocols below the transition temperature is generally found with the two branches merging at the transition point. In the case of spin-glass models, this low-temperature divergence is strongly enhanced in the presence of strong structural and magnetic frustration, which usually results from lattice distortions, structural disorder and the competition of the coexisting ferromagnetic and antiferromagnetic exchange interactions [76].

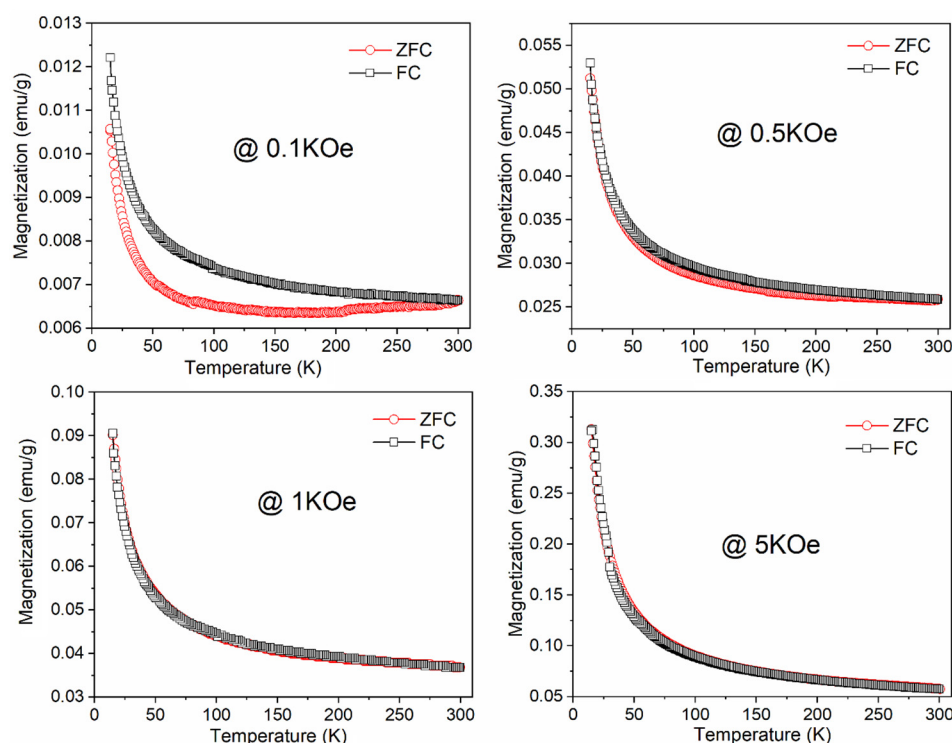


Figure 6. Magnetization versus temperature obtained in the ZFC and FC modes for applied fields of 0.1 kOe, 0.5 kOe, 1 kOe, and 5 kOe (where @ denotes the applied magnetic field).

The linear fit of the inverse magnetic susceptibility $\chi^{-1}(T)$ is illustrated in Figure 7. To analyze the $1/\chi$ vs. T curve, the Curie–Weiss equation was used [77]:

$$\chi^{-1} = \frac{T - \theta}{C} \quad (1)$$

with C signifying the Curie constant and θ representing the Weiss temperature. Concerning the effective magnetic moment per Fe ion (μ in μ_B/Fe), its estimation is determined from the Curie constant using the relation [77]

$$C = \frac{N\mu^2}{3k_B} \quad (2)$$

The linear fit of the χ^{-1} vs. T curve follows the Curie–Weiss law, giving a Weiss temperature $\theta = -9.64$ K and a Curie constant $C = 1.28 \times 10^{-3}$ (emu.K/g.Oe). The negative sign of θ provides definitive evidence for the predominance of short-range antiferromagnetic (AFM) exchange interactions between the coexisting transition-metal ions ($\text{Fe}^{2+}/\text{Fe}^{3+}$) embedded within the amorphous network [78]. This behavior is structurally rooted in the exceptionally high loading of industrial slag. By forcing an immense concentration of network modifiers and intermediates into the matrix baseline, the interstitial free volume of the glass framework is severely compromised. This profound structural compaction forces

adjacent iron cations into close spatial proximity, continuously activating anti-parallel super-exchange pathways via Fe-O-Fe linkages [79].

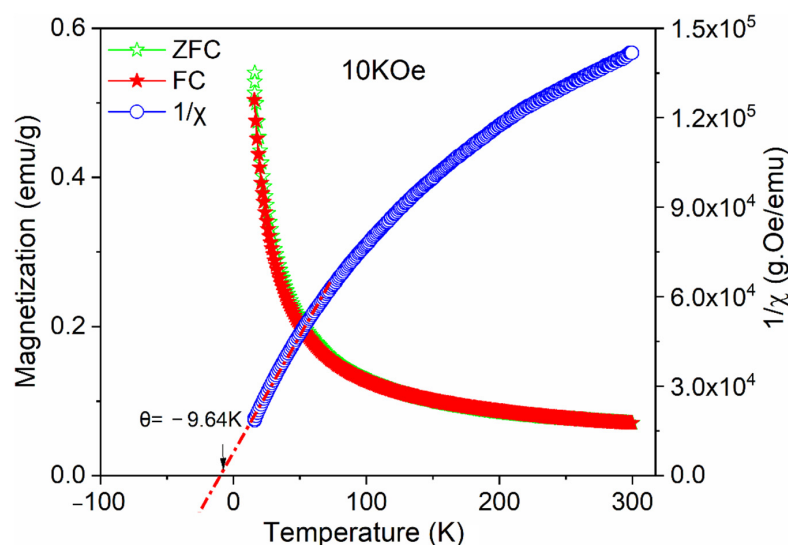


Figure 7. Temperature-dependent magnetization in both the ZFC and FC modes of the LiBS sample at $H = 10$ kOe, as well as the inverse of susceptibility $1/\chi$. The red dotted line represents the linear fit used to determine the Weiss temperature (θ).

The obtained effective magnetic moment per iron ion was determined to be $\mu_{\text{eff}} = 1.80 \mu_B$. This experimental value is exceptionally low compared to the theoretical spin-only moments expected for isolated high-spin iron species, which stand at $4.90 \mu_B$ for Fe^{2+} ($3d^6$) and $5.92 \mu_B$ for Fe^{3+} ($3d^5$) [80,81]. In the context of heavily waste-loaded transition-metal glasses, this unusually suppressed magnetic moment reveals a pronounced spin-masking effect. Due to the dense crowding of metallurgical slag within the amorphous network, the iron ions do not behave as magnetically isolated centers [82]. However, as the system is cooled to approach the freezing point, strong AFM super-exchange couplings among neighboring spins cause the great majority of the $\text{Fe}^{2+}/\text{Fe}^{3+}$ spins to be aligned in an anti-parallel manner. The cancelation of the magnetic contribution of such coupled spins means that a considerable fraction of the overall magnetization is not detected macroscopically.

3.5. Structure–Property Correlation: From Disordered Network to Frustrated Magnetic Behavior

The magnetic behavior of the LiBS glass can be directly correlated with the progressive reduction in local structural symmetry induced by the heavy incorporation of EAFS. In an ideal borate network, the $[\text{BO}_3]$ triangles and $[\text{BO}_4]$ tetrahedra possess relatively well-defined local geometry. Nonetheless, the concurrent addition of high amounts of $\text{Fe}^{2+}/\text{Fe}^{3+}$, Ca^{2+} , Si^{4+} , Mg^{2+} , and Mn^{2+} ions results in considerable deviations in terms of bond lengths and angles, causing a wide range of local environments to develop. Such structural disorder is a characteristic feature of multicomponent oxide glasses and has frequently been associated with symmetry breaking at the short- and medium-range scales [33,36].

The lack of local symmetry was observed in the form of marked broadening and overlapping of the FTIR and Raman bands due to different nonequivalent sites in the structure [83,84].

From a magnetic standpoint, these local distortions modify the Fe-O-Fe super-exchange pathways responsible for magnetic coupling. According to the Goodenough–Kanamori rules, the exchange interaction strength is highly sensitive to both the Fe-O bond distance and the Fe-O-Fe bond angle [85–87]. Therefore, the structural asymmetry generated within the

amorphous network produces a statistical distribution of exchange constants rather than a single well-defined interaction. Similar behavior has been reported in iron-containing disordered oxides, where random variations in local geometry generate competing magnetic interactions and magnetic frustration [37,40]. Consequently, neighboring iron ions experience competing magnetic couplings, leading to the coexistence of locally favored antiferromagnetic and weakly uncompensated magnetic regions.

This interpretation is supported by the magnetic measurements. The negative Weiss temperature ($\theta = -9.64$ K) shows that there are more antiferromagnetic interactions, and the very low effective magnetic moment ($\mu_{\text{eff}} = 1.80 \mu_B$) indicates that the magnetic moments cancel out each other effectively by having anti-parallel spins. Furthermore, the pronounced ZFC-FC bifurcation observed at low magnetic fields indicates the presence of magnetic frustration and spin freezing in disordered magnetic systems [39,40]. These observations collectively suggest that the degree of local symmetry breaking governs the distribution of magnetic exchange interactions and ultimately stabilizes the spin-glass-like state. Therefore, in the present glass system, the reduction in local structural symmetry acts as the microscopic origin of the observed frustrated magnetic behavior, consistent with the widely accepted picture of spin-glass formation in structurally disordered materials [37,39].

The relationship between the local structure framework and physical properties of the LiBS glass is an important landmark in the utilization of industrial waste to produce functional material. On an atomic scale, the large amount of 80 parts of metallurgical slag led to the modification of the fundamental lithium borate baseline lattice towards a tightly bound lithium borate structure. This topological change results from a strong structural association between the slag elements. Here the introduction of divalent Ca^{2+} and mixed valence iron ($\text{Fe}^{2+}/\text{Fe}^{3+}$) cations in the system helped fill the free volumes. The heavy metallic cations not only aided in compensating the charges for the $[\text{BO}_4]$ structural units but also helped introduce NBOs. These factors significantly contributed towards making the lattice much more densely packed. At the macro level, such a reinforcing effect clearly determines the outstanding stability of the glass, which can be seen in the high glass transition temperature ($T_g = 465^\circ\text{C}$). What is unique is that such restrictions and densification lead to immediate proximity between the incorporated iron ions. Thus, the reduction in the distance between the ions leads to the constant activation of strong anti-parallel Fe-O-Fe super-exchange interactions. Such a combination eventually results in a suppression of the effective magnetic moment at an exceptionally low level, $\mu_{\text{eff}} = 1.80 \mu_B$, due to a significant masking effect, while the magnetic frustration ensures disordered spin-glass behavior of the sample.

While the macroscopic ZFC-FC magnetization bifurcation and calculated frustration parameters strongly suggest a frustrated, spin-glass-like magnetic state, we acknowledge that direct probing of the local iron environment and exact $\text{Fe}^{2+}/\text{Fe}^{3+}$ ratios (e.g., via Mössbauer spectroscopy or X-ray photoelectron spectroscopy) is required to definitively resolve the underlying short-range magnetic exchange mechanisms. This structural-magnetic correlation remains a key perspective for future investigations.

4. Conclusions

In summary, the 50Li₂O-50B₂O₃-80 wt.% slag system was prepared by the melt-quenching method. Characterization techniques such as XRD, DSC, density measurement, FTIR, and Raman spectroscopy were used to give a full explanation of the structural arrangement at different length scales. The synthesized glass showed remarkable thermal stability with a relatively high glass transition temperature ($T_g = 465^\circ\text{C}$). The increased rigidity is a direct result of the structural strengthening effect of the major slag constituents on the multicomponent vitreous framework. Magnetic investigations revealed an intriguing

highly frustrated magnetic structure embedded in a structurally amorphous environment. The temperature dependence of magnetic susceptibility exhibits clear Curie–Weiss behavior at low temperatures, indicating a negative paramagnetic Curie–Weiss temperature. This proves the absolute predominance of antiferromagnetic super-exchange interactions through Fe–O–Fe pathways.

Additionally, the extremely high density of mixed-valent iron ions ($\text{Fe}^{2+}/\text{Fe}^{3+}$) leads to a strong spin-masking effect, resulting in an extremely low experimental effective magnetic moment of $\mu_{\text{eff}} = 1.80 \mu_B$. The negative Weiss temperature together with the ZFC–FC bifurcation indicate a highly frustrated magnetic state that may evolve toward spin-glass-like behavior at low temperature.

These results are a double success, providing not only an environmentally friendly approach to metallurgical solid waste but also an efficient way to synthesize magnetically active amorphous materials.

On an industrial scale, these magnetically active glasses can be easily processed into fine powders and incorporated as functional micro-fillers in polymer or ceramic composites for electromagnetic interference (EMI) shielding or low-cost magnetic sensor applications. Furthermore, the synthesis temperature of 1100 °C is fully compatible with standard industrial glass furnaces and rotary kilns, facilitating seamless scalability without requiring specialized ultra-high-temperature infrastructure.

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Abbreviations

The following abbreviations are used in this manuscript:

AFM	Antiferromagnetic
DSC	Differential Scanning Calorimetry
EAFS	Electric Arc Furnace Slag
FC	Field-Cooled
FTIR	Fourier-Transform Infrared Spectroscopy
NBO	Non-Bridging Oxygen
SEM	Scanning Electron Microscopy
T _c	Crystallization Temperature
T _g	Glass Transition Temperature
TM	Transition Metal
VSM	Vibrating Sample Magnetometer
XRD	X-Ray Diffraction
ZFC	Zero-Field-Cooled

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