

Effect of Red Mud Loading on the Thermal and Mechanical Performance of Polypropylene Composites

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Abstract

Waste red mud (RM) serves as a low-cost and sustainable filler material in a polypropylene (PP) matrix. This study investigate the effects of different red mud contents on the dynamic mechanical and thermal properties of polypropylene-red mud (RM-PP) composites where red mud levels range from 0% to 40% (based on weight percentage). Fourier Transform Infrared Spectroscopy (FTIR) was conducted for verifying the presence of both polypropylene and red mud in composite samples. The dispersion of red mud within the polypropylene matrix was examined through Scanning Electron Microscopy (SEM). X-ray Diffraction (XRD) analysis was performed to evaluate the crystalline structure of the composites. Thermogravimetric Analysis (TGA) assessed the thermal stability of the polypropylene-red mud composites, specifically determining their resistance to thermal degradation; notably, the composite containing 40% red mud exhibited excellent thermal stability. Differential Scanning Calorimetry (DSC) was utilized to compare the melting temperatures (T_m) of the composites, showing that the melting temperatures of the polypropylene matrix increased following the incorporation of red mud, indicating strong interactions between polypropylene and red mud. Additionally, Dynamic Mechanical Analysis (DMA) results demonstrated a decrease in storage modulus with increasing red mud content, while XRD findings confirmed that crystallinity was increased with red mud concentration.

Keywords: Polypropylene (PP), Red mud (RM), Polypropylene-red mud (RM-PP) composites, Thermal behavior, Storage modulus.

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Received Date: March 17, 2026

Accepted Date: March 27, 2026

Published Date: May 05, 2026

Citation: Sourav Basu, Anupam Maiti, Rajib Gupta, Sujan Krishna Samanta, Soumya Mukherjee, Akshay Kumar Pramanick, Sourav Debnath. Effect of Red Mud Loading on the Thermal and Mechanical Performance of Polypropylene Composites. Journal of Polymer & Composites. 2026; 14(Special Issue 3): S161–S169p.

INTRODUCTION

The increasing demand for eco-friendly polymers is driven by environmental concerns and the depletion of fossil resources. Reusing industrial waste such as red mud offers significant environmental and economic advantages. Red mud is invented as byproduct during alumina production via the Bayer process [1]–[3]. About one tonne per tonne of alumina is produced annually worldwide, amounting which 120–150 million tonnes are red mud [1, 4]. Its highly alkaline and toxic nature, containing heavy metals and radioactive elements, makes disposal a major challenge, with common methods including ocean dumping, lagoon storage, and dry stacking [2, 5, 6].

Due to its variable chemical and mineralogical composition and fine particle size, red mud has been explored for applications in construction

materials [4,5], catalysis [6], geopolymers [7, 8, 9], ceramics [10], iron and steel production [9], and metal and polymer matrix composites [3]. Its reuse supports environmental protection, reduces storage issues, lowers aluminum production costs, and enables recovery of valuable earth elements like iron, aluminum, titanium etc.

Research interest in red mud has grown rapidly since 2010, driven by circular economy initiatives, though further studies are required for large-scale utilization [11, 12, 13]. Recently, red mud has gained attention as a low-cost filler in polymer composites. Studies on polypropylene-red mud (RM-PP) composites report improved stiffness and thermal properties with increasing red mud content, although impact resistance and ductility may decrease at higher loadings [12]–[17].

This study aims to investigate waste red mud as a sustainable and cost-effective filler in a polypropylene matrix, focusing on its effects on the dynamic mechanical and thermal properties of the composites and its potential to enhance overall performance while reducing environmental impact.

EXPERIMENTAL

In this study, red mud with density 0.9 g/cm³, a melt flow index (MFI) of 15 gm per 10 minutes (230°C, 2.16 kg) and a heat deflection temperature 95°C (0.45 MPa, unannealed) is being considered.

Development of Composites

Red mud was heat-treated at temperature ranging 15°C–170°C for remove hydrates followed by dry-ground to obtain homogeneous powders with particle sizes of 38–250 µm. The solid components were mechanically premixed for 15 min using an LB-5601 liquid–solids batch blender. Polypropylene (PP) composites with varying red mud (RM) contents were prepared using a Microsan extruder operated at 170–220°C, 25–30 bar with rotation of 23 rpm. The extrudates were dried at 80°C for a day long in vacuum oven followed by injection-moulded at 170–220°C, 90–120 bar at same rotation. Five composite formulations were prepared with PP-RM weight ratios of 100/0, 90/10, 80/20, 70/30, and 60/40.

Characterization Methods

The developed polymer composites were characterized by FTIR (model no. PerkinElmer Spectrum, 600–4000 cm⁻¹). Surface texture was examined employing “Zeiss EVO LS scanning electron microscope”. XRD analysis was performed using “Panalytical X’PERT PRO diffractometer with CuKα radiation at 45 kV and 40 mA” under ambient conditions. DMA was conducted using a PerkinElmer DMA 8000 in single cantilever mode (50 µm amplitude, 1 Hz, 5°C/min, –50 to 150°C). TGA measurements were carried out on an EXSTAR TG/DTA 6300 Seiko instrument at 800°C at inert nitrogen. DSC analysis (8 ± 1 mg, n = 5) was performed using a “PerkinElmer Diamond DSC 8230 under inert atmosphere (50 mL/min)” at 10°C/min over 20–200°C, with data processed using Pyris software.

RESULTS & DISCUSSION

FTIR Analysis

Chemical bonds are identified in organic and inorganic materials using FTIR analysis, enabling compositional analysis [15,16]. Figure 1 shows the FTIR spectra of PP-RM composites with varying red mud content.

The “main absorption bands” [17] in PP spectra were observed at 2918 cm⁻¹ (CH₂), 2951 cm⁻¹ (CH₃),

2839 cm⁻¹ (CH₂), 1457 cm⁻¹ (CH₂), 1376 cm⁻¹ (CH₃), 1359 cm⁻¹ (CH₂–CH), 1330 cm⁻¹ (CH₂–CH), 998 cm⁻¹ (CH₃), 973 cm⁻¹ (CH₃, C–C), 841 cm⁻¹ (C–H), 808 cm⁻¹ (C–C), and at 1304, 1223, 1261, 1168, and 1154 cm⁻¹ for CH₃, CH₂, and CH groups, respectively [17]. The intensities of PP bands are decreased with the increment of red mud into polypropylene matrix.

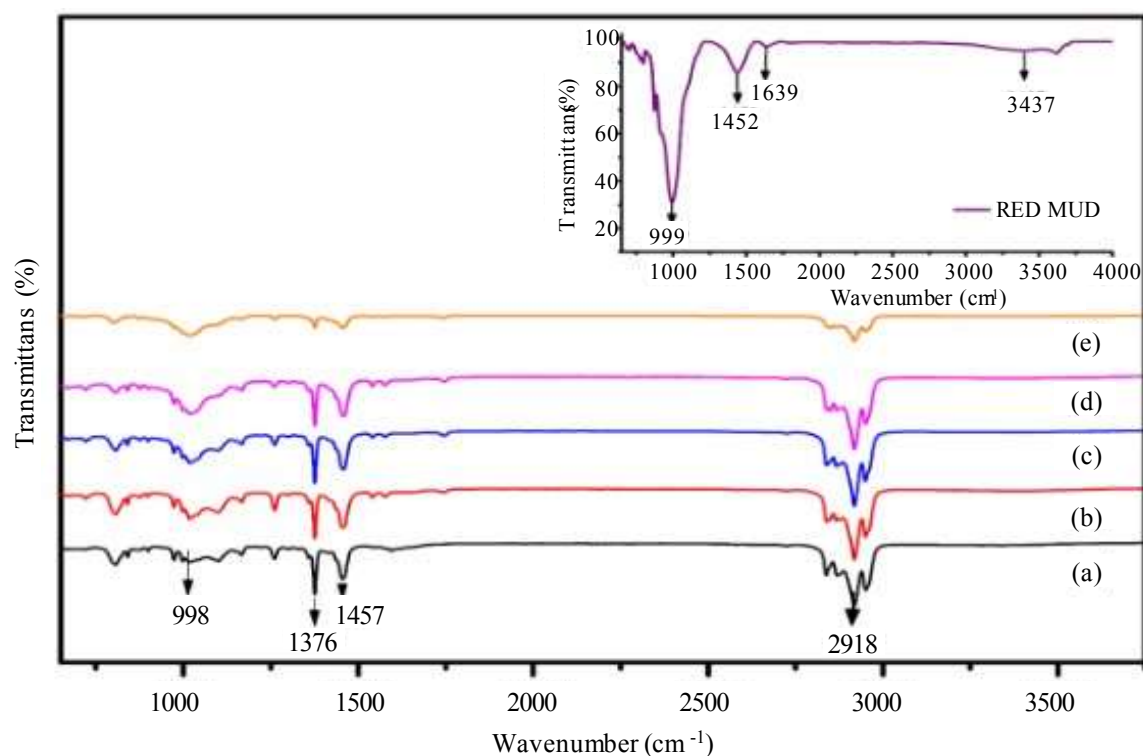


Figure 1. FTIR spectra of (a) polypropylene, (b) 10 percentage, (c) 20 percentage, (d) 30 percentage, (e) 40 percentage RM-PP composites

In the FTIR spectrum of RM, absorption bands appeared at 3437, 2920, 2853, 1745, 1639, 1578, 1560, 1542, 1452, 1402, 995, 802, and 654 cm^{-1} [18]. Hydrogen-bonded surface OH groups are found out 3437 cm^{-1} . Symmetric and asymmetric CH_2 stretching are observed at 2920 cm^{-1} and 2853 cm^{-1} from organic hydrocarbons present in red mud. The absorption band at 1745 cm^{-1} is associated with the C=O double bond. The band at 1639 cm^{-1} is related to H–O–H bending vibrations from structural water in aluminosilicates. The band at 1452 cm^{-1} likely arises from carbonate components such as calcite. The “band at 999 cm^{-1} can be assigned to Si–O–Si stretching vibrations” [19][20][21]. FTIR results confirmed “the incorporation of red mud into the polypropylene matrix” and the occurrence of interactions between the two components.

Surface Morphology

Surface morphology of PP-RM composites of various compositions are shown in figure 2. Red mud particle clusters are dispersed within the polypropylene matrix, and their agglomeration increases with higher red mud content (Figure 2b–d). As the concentration of red mud rises, formation of agglomerates becomes more pronounced, which contributes to a reduction in the storage modulus. These observations from SEM images are consistent with the DMA results.

XRD

XRD patterns of RM, neat PP, and PP composites of various compositions are shown in figure 3. The RM pattern reveals a multiphase composition, consistent with literature reports, with major phases identified as “quartz (SiO_2), dicalcium silicate (Ca_2SiO_4), hematite (Fe_2O_3), calcite (CaCO_3), rutile (TiO_2), sodium aluminum silicate (Na(AlSiO_4)), gibbsite (Al(OH)_3), and goethite (FeO(OH)), identified using the ICDD database in X’PertHighScore software” [22]. Neat PP exhibits characteristic crystalline peaks at $2\theta = 13.9^\circ$ and 16.7° (Figure 3a), in agreement with reported data [23]. With increasing RM content, PP peak intensities decrease while additional peaks from RM minerals appear, indicating enhanced crystallinity of the PP matrix. Since polymer crystallinity strongly affects thermal properties, such changes can contribute to improved thermal performance [24–26].

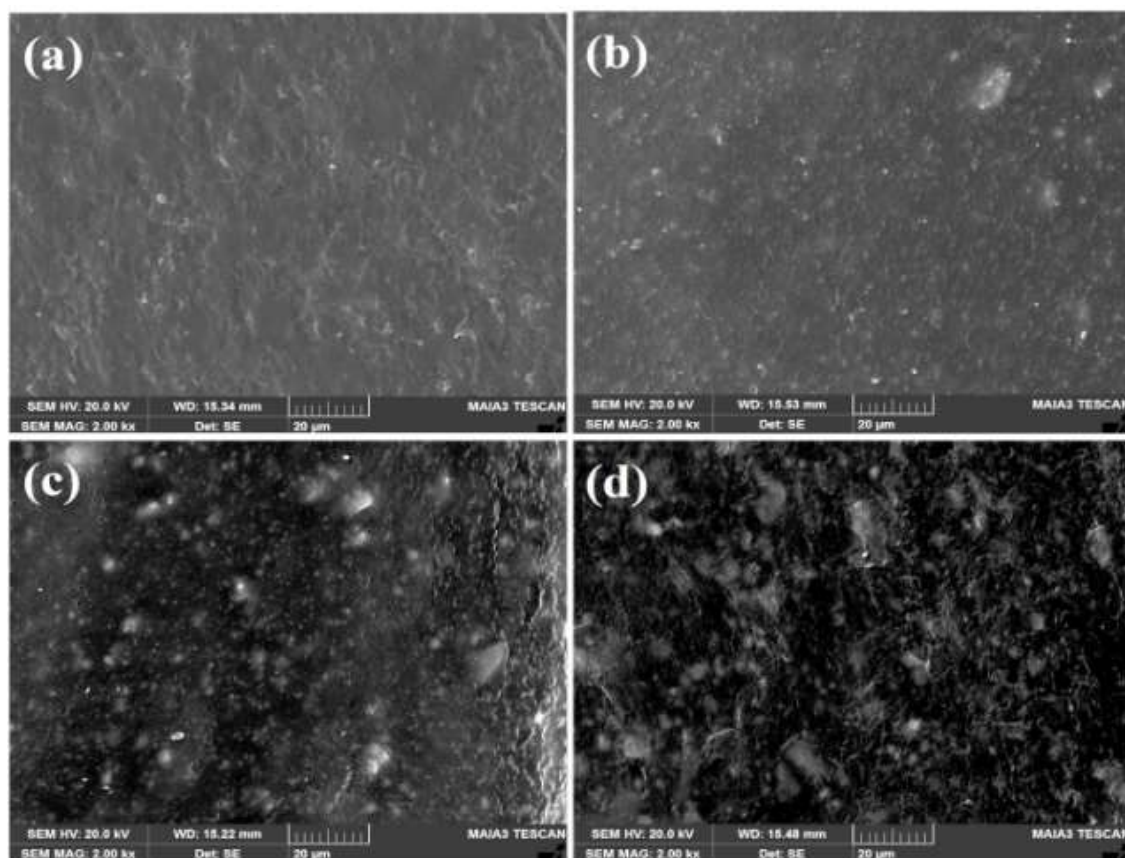


Figure 2. Surface morphology (SEM) of (a) polypropylene, (b) 10%, (c) 30%, (d) 40% RM-PP composites

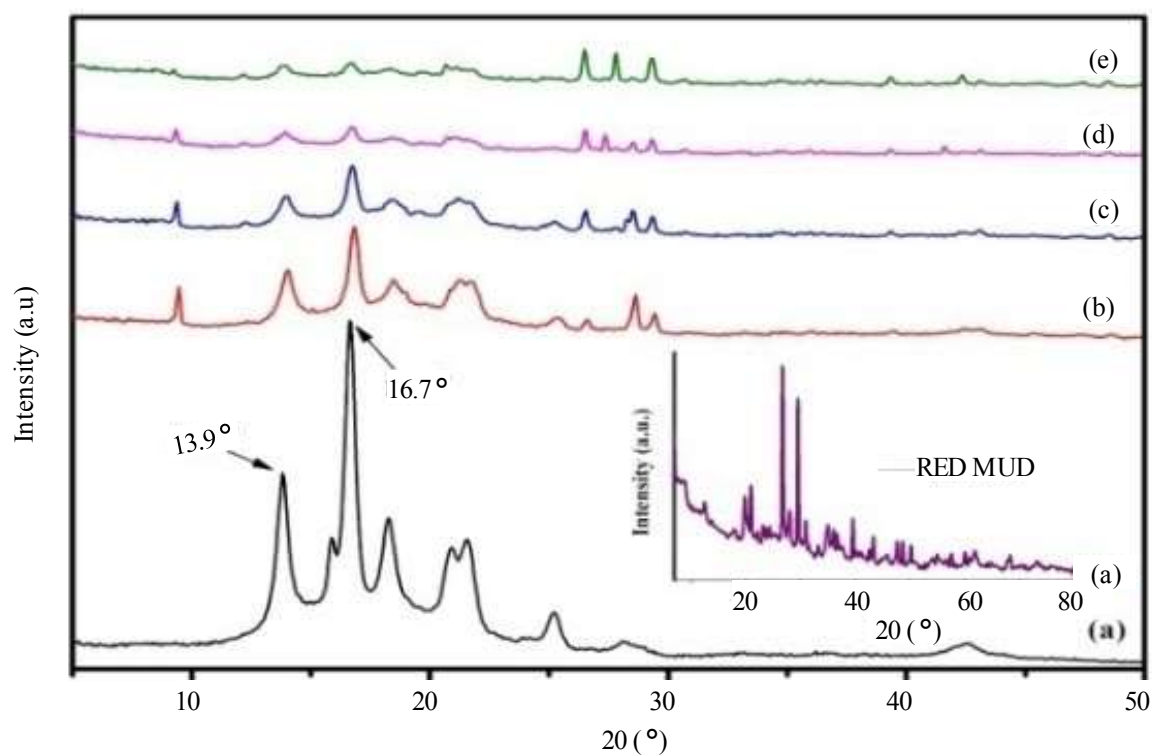


Figure 3. XRD patterns of (a) polypropylene, (b) 10%, (c) 20%, (d) 30%, (e) 40% RM-PP composites

Following XRD analysis, the crystallinity percentage of the red mud–reinforced polypropylene composites was calculated using universal equation describe elsewhere [27, 28, 29]. Observed that, percentage of crystallinity increased with higher red mud content. The crystallinity values were 90.29% for red mud, 80.04% for polypropylene, 80.18% for the 10% PP-RM composite, 83.75% for the 20% PP-RM composite, 87.34% for the 30% PP-RM composite, and 94.94% for the 40% PP-RM composite.

The degree of crystallinity in the material increased proportionally with the rising concentration of highly crystalline RM within the matrix. This trend is further supported as observed from additional peaks in the XRD spectrum as the RM content increased, indicating a greater level of crystalline structure.

DMA

Figure 4 presents the temperature-dependent storage modulus of polypropylene and RM-PP composites containing 10%, 20%, 30%, and 40% red mud. The storage modulus (E') of the red mud-filled composites was lower than that of pure PP, particularly at lower temperatures. Storage modulus is decreased due to increment of elasticity imparted by the presence of red mud. At 25°C, the storage modulus of polypropylene was measured at 6.28×10^8 Pa, whereas the composite containing 40% red mud exhibited a storage modulus of only 2.60×10^8 Pa at the same temperature—a reduction of approximately 58%. This decline is attributed to the increased mobility of polymer chains under external force [30, 31, 32].

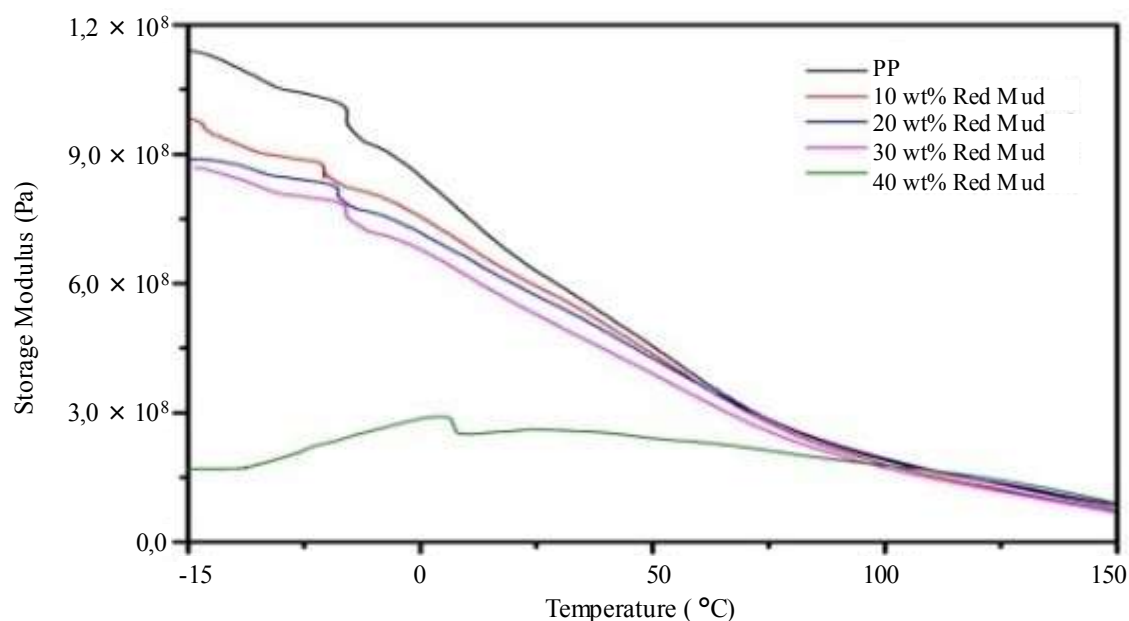


Figure 4. Variation of storage modulus for PP-RM composites with red mud content

TGA

The thermogravimetric analysis (TGA) curves of red mud, neat polypropylene (PP), and RM-PP composites containing 10%, 20%, 30%, and 40% red mud. Neat polypropylene shows a single major degradation step, with rapid weight loss occurring between approximately 400 and 480°C, associated with the thermal decomposition of the polymer. In contrast, red mud exhibits high thermal stability over the entire temperature range, with only minor weight loss attributed to the removal of adsorbed moisture and dehydroxylation of mineral phases.

For the PP-RM composites, thermal degradation occurs at slightly higher temperatures compared to neat polypropylene, indicating improved thermal stability with red mud incorporation. As the RM content increases, the onset of degradation shifts toward higher temperatures and the residual char content at elevated temperatures increases significantly. The remaining mass at 800°C corresponds to

the inorganic fraction of red mud, which acts as a thermally stable residue. These results suggest that red mud restricts polymer chain mobility and acts as a thermal barrier, thereby enhancing the thermal stability of polypropylene composites [28]-[32].

DSC

The DSC melting curves for polypropylene and PP-RM composites with different mixing ratios are shown in Figure 5. Polypropylene and polypropylene with 10%, 20%, 30%, and 40% red mud had melting temperatures (T_m) of 169.08, 169.62, 169.65, 169.77, and 171.45°C, respectively. When RM is added, T_m shifts toward higher temperatures, indicating better thermal behavior as well as favorable compatibility and interaction between polypropylene and red mud.

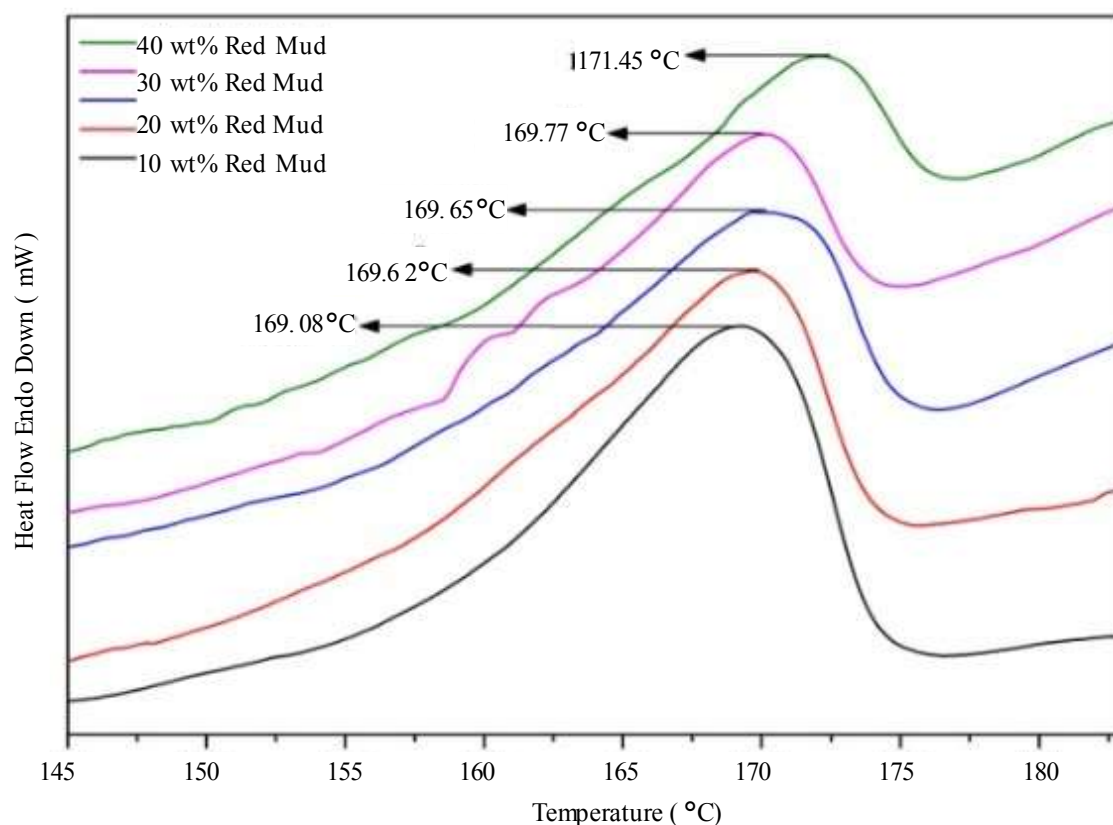


Figure 5: DSC curves for various PP-RM composites

CONCLUSIONS

Polypropylene based red mud composites were developed successfully and various properties like thermal, structural, and dynamic mechanical have found out for each developed composite. The distinct crystal structures and mechanical characteristics of red mud influenced the thermal behavior and dynamic mechanical performance of the composites. SEM analysis showed increased agglomeration with higher red mud content, and DMA results indicated that this agglomeration contributed to greater elasticity in the composites. FTIR analysis confirmed the presence of both polypropylene and red mud in the materials, revealing a decrease in polypropylene peak intensities and the appearance of new peaks associated with red mud.

XRD analysis demonstrated that the crystallinity of the composites increased as red mud content rose, supported by the emergence of additional peaks in the diffraction patterns. The upward shift in melting temperature (T_m) observed in the DSC results further indicated good compatibility and interaction between polypropylene and red mud. TGA findings showed that incorporating low-cost red mud enhanced the thermal stability of polypropylene, which naturally has limited thermal resistance.

From an economic standpoint, using waste materials like red mud lowers trash disposal costs while creating chances for new industrial applications like automobile structures, packaging, corrosion-resistant components, and so forth. All things considered, adding red dirt to polypropylene has both financial and environmental benefits. For applications where elasticity and thermal stability are

important, polypropylene based red mud composites are suitable and costeffective. Furthermore, polypropylene–red mud composites with a 40% red mud content can gradually be used to make a variety of furnace interior components such as resistant bricks.

AUTHOR CONTRIBUTIONS

The first author and the second author contributed equally to this work and share first authorship.

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