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Enhancing Mechanical, Thermal, and Rheological Properties of Recycled Polyethylene via Nano-Calcium Carbonate Reinforcement

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ABSTRACT

The increasing global demand for polyethylene has also led to a large amount of waste generated from this polymer, posing numerous environmental and economic challenges. Due to its high chemical stability, polyethylene waste is difficult to decompose and can remain in the environment for decades, leading to soil and water pollution.This study investigates the effect of incorporating nano-calcium carbonate ($CaCO_3$) into recycled polyethylene (rPE) composites. Due to extensive industrial use, polyethylene constitutes a significant portion of plastic waste, and its recycling often leads to reduced mechanical and thermal performance. The aim of this work was to improve the quality and durability of rPE by nanoparticle reinforcement, enabling efficient reuse in multiple recycling cycles.rPE samples containing 0–10 wt% $CaCO_3$ nanoparticles were prepared via melt blending and twin-screw extrusion. Mechanical (tensile strength, Young's modulus, hardness), thermal (oxidation induction time, thermal expansion, DSC, TGA), and rheological (melt flow rate) tests were performed, complemented by microscopic analysis. Statistical analyses were conducted using one-way ANOVA and Duncan's test.Results showed that 5 wt% $CaCO_3$ yielded optimal performance, increasing tensile strength and hardness, improving thermal stability, reducing thermal expansion, and maintaining favorable melt flow properties. Post-recycling tests indicated that nanoparticle-filled samples retained ~90% of their original mechanical properties after three recycling cycles, whereas neat rPE retained only 75%. Microscopy confirmed uniform nanoparticle dispersion at optimal loadings, while higher concentrations led to agglomeration and reduced performance.These findings demonstrate that nano-$CaCO_3$ reinforcement is a cost-effective and environmentally beneficial approach to enhance recycled polyethylene properties, supporting sustainable polymer recycling and circular economy initiatives.

INTRODUCTION

Polyethylene (PE) is one of the most widely used polymers in industrial applications due to its chemical resistance, flexibility, and ease of processing. However, its mechanical properties and long-term durability are often limited, particularly in demanding applications such as pressure and corrugated pipes. The development of polymer nanocomposites, reinforced with nanoparticles, offers a promising approach to overcome these limitations. Calcium carbonate ($CaCO_3$) nanoparticles, with high surface area and strong interfacial interactions with polymer chains, can significantly enhance mechanical strength, thermal stability, and processability of PE-based materials [1, 2, 3]. Previous studies have shown that incorporating $CaCO_3$ nanoparticles into medium-density polyethylene (MDPE) increases resistance to penetration and indentation, with optimal improvements observed at around 5 wt% loading [1]. Similarly, in polypropylene/poly(ethylene terephthalate) (PP/PET) blends, varying $CaCO_3$ concentrations can influence morphology, crystallization behavior, and mechanical properties [2]. In high-density polyethylene (HDPE) nanocomposites, $CaCO_3$ nanoparticles combined with elastomers such as POE have demonstrated enhanced tensile and impact properties [3].The integration of recycled HDPE (rHDPE) into pipe production supports sustainability and circular economy practices by reducing reliance on virgin materials and enabling end-of-life reuse of pipes [4, 5]. Life Cycle Assessment (LCA) studies indicate that the recycled content notably affects the

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environmental footprint of pipe-grade resins [5]. Mechanical and processing performance of recycled double-wall corrugated HDPE/PPR pipes can be further improved using compatibilizers such as maleic anhydride-grafted polyethylene (POE-g-MAH) alongside $CaCO_3$ nanoparticles, enhancing interfacial adhesion, dispersion, tensile and flexural performance, and impact resistance [6]. Studies on polymer matrices other than PE, such as PVC, have also shown that surface-modified $CaCO_3$ nanoparticles can improve thermal and mechanical properties of nanocomposites, highlighting the importance of nanoparticle surface treatment and dispersion [7]. Moreover, investigations into HDPE filled with $CaCO_3$ (in the micro to nano range) suggest that while stiffness and thermal stability may increase, careful control of filler size, content, and dispersion is crucial to avoid brittleness or processing issues [8, 9]. Given the synergistic role of $CaCO_3$ nanoparticles in reinforcing polymer matrices and the growing importance of recycling and circular economy in pipe manufacture, this study aims to systematically investigate the effects of $CaCO_3$ nanoparticle incorporation on the mechanical performance, processability, and recyclability of PE pipes, providing insights into optimizing nanocomposite design for sustainable, high-performance polymer piping systems [10]. The use of calcium carbonate ($CaCO_3$) nanoparticles as reinforcing fillers in polymeric matrices has attracted considerable attention due to their cost-effectiveness, availability, and ability to improve the mechanical and thermal performance of polymers used in pipe applications [11]. Several studies have reported that $CaCO_3$-filled polyethylene systems exhibit improved stiffness, penetration resistance, and dimensional stability, which are critical requirements for pressure pipe applications [12]. Previous investigations demonstrated that the incorporation of $CaCO_3$ nanoparticles into polyolefin matrices used for piping systems can enhance tensile properties, thermal stability, and crystallization behavior, provided that an optimal filler concentration and homogeneous dispersion are achieved [13]. Furthermore, recent studies on recycled HDPE pressure pipes indicate that rHDPE can partially replace virgin PE in pipe manufacturing within a circular economy framework, although mechanical and rheological properties must be carefully controlled to ensure long-term pipe performance [10]. This study focuses on examining the influence of calcium carbonate nanoparticles on the recyclability of polyethylene pipes. Due to the large amount of polyethylene pipe waste, environmental pollution and the price of pipe raw materials, these materials can be returned to the production cycle by studying and examining them.The investigation primarily aims to assess the physical and mechanical characteristics, along with the recyclability performance of polyethylene pipes reinforced with calcium carbonate nanoparticles.

MATERIALS AND METHODS

Materials

The materials and equipment used in this study were purchased . Recycled polyethylene (rPE) was derived from HDPE (PE100) pipes, while nano-calcium carbonate (nano-$CaCO_3$, 50-70 nm, 98.5%) was supplied by Hooman Chimie Pars. Polyethylene (HDPE, PE100) prepared from Shazand petrochemical Company.twin-screw extruder was purchased from Coperion, Thermo Fisher Scientific.

Preparation of Recycled Polyethylene

HDPE pipes were first cut into standard segments, then shredded and thoroughly washed to remove contaminants. The cleaned material was subsequently melted at 180–220 °C using extrusion or injection molding. The molten polymer was pelletized to produce recycled polyethylene (rPE) granules for further processing.

Preparation of Nano-$CaCO_3$ Composites

Nano-sized calcium carbonate ($CaCO_3$) particles, with an average diameter of 10–50 nm, were incorporated into the polyethylene matrix to enhance mechanical and thermal performance. The nanoparticles were first dispersed in the polyethylene resin using either high-shear mechanical stirring to ensure uniform distribution and minimize agglomeration. Recycled polyethylene (rPE) was then combined with the dispersed nanoparticles at various weight fractions (1, 2, 5, and 10 wt%). The resulting mixtures were thoroughly blended to achieve a homogeneous composite and subsequently pelletized to produce uniform rPE/$CaCO_3$ granules suitable for downstream processing, including extrusion or injection molding..

Compounding and Extrusion

Nano-$CaCO_3$ particles and recycled polyethylene (rPE) were compounded using a co-rotating twin-screw extruder at approximately 200 °C to achieve uniform melting and dispersion. The extrudates were cooled to room temperature, pelletized, and subsequently molded into standard test specimens suitable for mechanical and thermal characterization.

Mechanical and Physical Testing

Mechanical tests included tensile strength, Young's modulus, elongation at break, and Shore D hardness. Thermal behavior was assessed via OIT, TGA, DSC, and coefficient of thermal expansion. Rheological properties were measured via MFR. Standard test methods included ASTM D638, ISO 527, ISIRI 17140. Post-recycling tests were conducted after three cycles to evaluate property retention. Data were analyzed using SPSS 24, one-way ANOVA, and Duncan's test.

RESULTS AND DISCUSSION

Tensile Properties

Figure 1 shown Tensile strength and Young's modulus increased based on $CaCO_3$ percent of four samples. The $CaCO_3$ content up to 5 wt%, while elongation at break slightly decreased. Beyond 5 wt%, tensile strength declined due to nanoparticle agglomeration. Statistical analysis confirmed that 5 wt% $CaCO_3$ was the optimal loading.

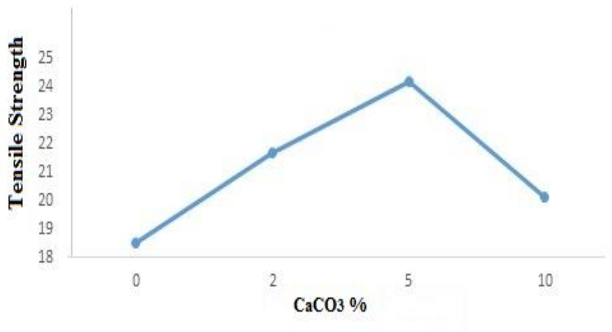

Fig. 1. Tensile strength diagram for rPE samples

Hardness

Shore D hardness increased with nanoparticle content, peaking at 5 wt% (70 Shore D) and slightly decreasing at 10 wt% due to agglomeration (Figure 2).

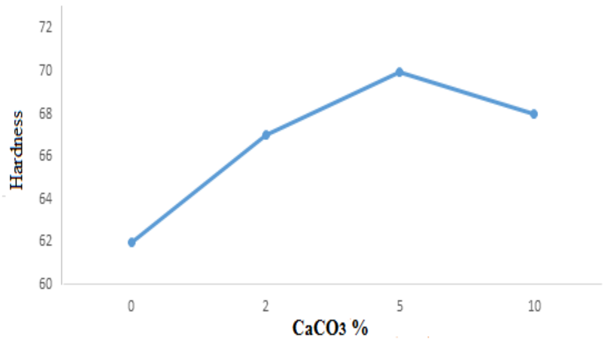

Fig. 2. Hardness test for rPE samples

Thermal Expansion

Figure 3 shows the coefficient of thermal expansion decreased with increasing nanoparticle content, reaching 115 µm/m°C at 5 wt%, indicating improved dimensional stability. However, an excessive increase in the nanoparticle content (10%) has the opposite effect due to particle aggregation and results in a relative increase in CTE.

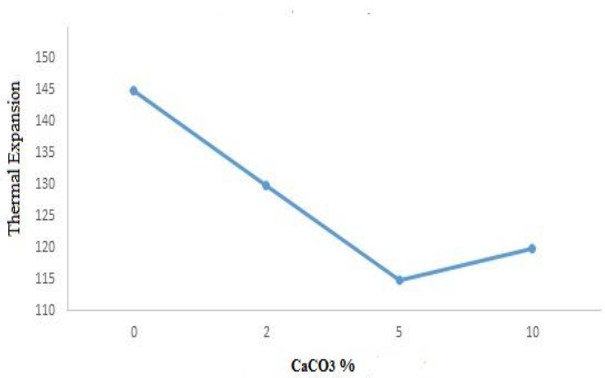

Fig. 3. Thermal expansion test diagram for rPE samples

Melt Flow Rate

MFR decreased with nanoparticle addition, reflecting higher viscosity and improved processability. In **Figure 4**, a relative increase in MFR (about 7.9) is observed, which could be due to:agglomeration of nanoparticles,reduction of effective particle-polymer contact area or creation of slip paths in the melt. This behavior is very common for polymer composites and nanocomposites.

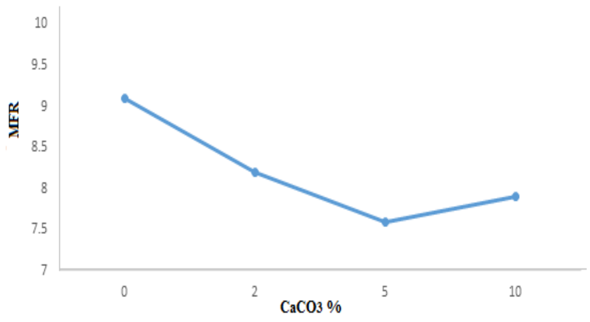

Fig. 4. Melt flow rate for rPE samples

Oxidation Induction Time (OIT)

OIT increased with $CaCO_3$ loading, with the 5 wt% sample showing maximum resistance to oxidative degradation. Nanoparticles act as a barrier to oxygen diffusion. However, at 10%, a relative decrease in OIT was observed, which can be attributed to particle aggregation and reduced antioxidant efficiency (Figure 5).

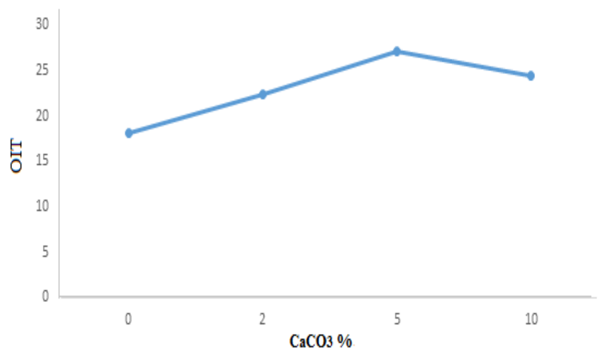

Fig. 5. Oxidation induction time test of rPE samples

Post-recycling data analysis

Recycled samples were retested after adding nanoparticles. The results showed that with the addition of nanoparticles, physical properties such as density and surface smoothness improved and mechanical properties such as hardness and tensile strength increased compared to the pure recycled sample.The optimal percentage of calcium carbonate nanoparticles is 5%.This percentage increases tensile strength, hardness, reduces thermal expansion and better controls viscosity.Increasing more than this amount reduces the uniformity of the structure and mechanical performance. Recycling with nano additives greatly restores the properties of the composite.

Statistical analysis of the tensile test

According to the results presented in Figure 6, the effect of different calcium carbonate ($CaCO_3$) nanoparticle contents on the mechanical properties of the samples was investigated. The results indicate that increasing the nanoparticle content from 0% to 5% leads to a significant improvement in tensile strength. The tensile strength increased from approximately 900 for the neat sample to about 1100 for the sample containing 2% nanoparticles and reached a maximum value of nearly 1250 at 5% $CaCO_3$ loading.This enhancement can be attributed to the uniform dispersion of nanoparticles within the polymer matrix and the improved stress transfer between the reinforcing phase and the matrix. The presence of an optimal amount of nanoparticles restricts the mobility of polymer chains, resulting in increased tensile strength and elastic modulus.However, when the nanoparticle content was further increased to 10%, a slight reduction in tensile strength (approximately 1150) was observed. This decrease is mainly associated with particle agglomeration and non-uniform dispersion, which generate stress concentration sites and weaken interfacial bonding. Nevertheless, the tensile strength at 10% loading remained higher than that of the neat sample.In addition, the hardness results exhibited a decreasing trend with increasing nanoparticle content. The hardness value decreased from approximately 530 for the unfilled sample to around 430 for the sample containing 10% $CaCO_3$. This behavior may be related to microstructural changes and the formation of localized discontinuities caused by particle aggregation.The one-way analysis of variance (One-Way ANOVA) confirmed that the differences among the samples were statistically significant at the 95% confidence level ($p < 0.05$). Furthermore, Duncan's post hoc test revealed that the sample containing 5% $CaCO_3$ nanoparticles exhibited significantly superior mechanical performance compared to the other groups.Overall, based on both statistical and mechanical analyses, a $CaCO_3$ nanoparticle content of 5% is considered the optimal loading for enhancing the tensile properties and overall mechanical performance of the composite.

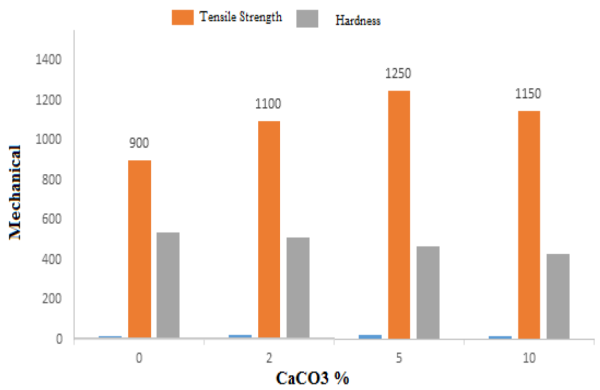

Fig. 6. Statistical analysis of the mechanical tests for rPE samples

Microscopic Analysis

The images were taken using a tension microscope, pixel ferber imagimg software, KEview camera, according to ISO 18553 and INSO20059 standards.**Figure 7** shows tension images confirmed uniform dispersion at 2–5 wt%, while agglomeration at 10 wt% caused reduced mechanical performance.

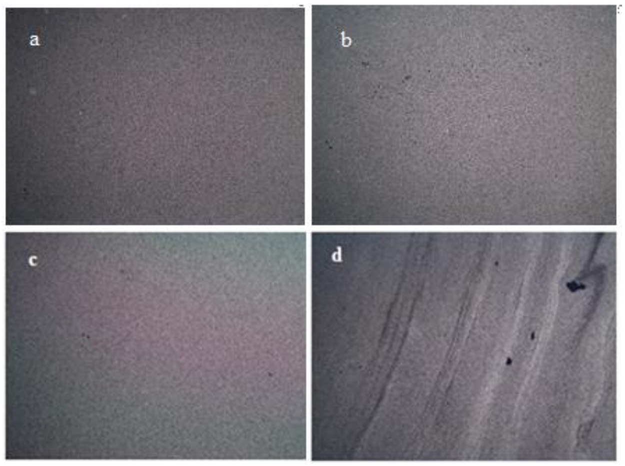

Fig. 7. Tension images of rPE a) 0% samples b) 2% samples c) 5% samples d) 10% samples

Thermal behavior of samples investigated by TGA and DSC tests

The thermal analysis test by TGA and DSC was performed to investigate the thermal stability and transition behavior of the samples. a) 0% nanopartciles has O.I.T=18.02 , b) 2% nanopartilces O.I.T=22.04 , c) 5% nanoparticles has O.I.T =27.01 and 10% nanopartciles has O.I.T=24.50. the 5% nanoparticles showed the highest OIT value, indicating its greater resistance to thermal and oxidative degradation. 10% nanoparticles can be due to particle aggregation and the creation of weaker areas in the polymer matrix, which accelerates the onset of degradation.The results showed that the addition of calcium carbonate nanoparticles ($CaCO_3$) increased the thermal stability and also improved the glass transition behavior. In the TGA test, the sample containing 5% nanoparticles showed a thermal degradation onset temperature about 20 degrees Celsius higher than the sample without nanoparticles.This indicates the inhibitory role of nanoparticles in preventing the rapid decomposition of the polymer matrix.In the DSC test, an increase in the glass transition temperature (Tg) was observed in the presence of nanoparticles.

This increase was due to the interaction between the polymer phase and the mineral phase, which limited the movement of polymer chains and increased the thermal resistance of the material.Overall, the results of the TGA and DSC tests showed that the addition of calcium carbonate nanoparticles, in addition to increasing thermal stability, also improved the thermal and structural properties of the material and could lead to increased durability and performance of composites under operational conditions (Figure 8).

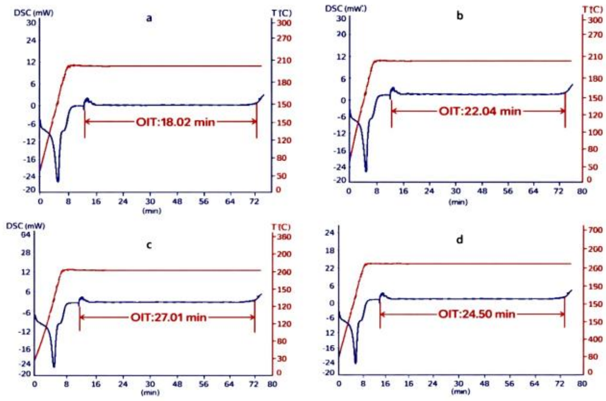

Fig. 8. DSC and OIT a)0% nanoparticles b) 2% nanoparticles c) 5% nano particles d)10 % nanoparticles

Analysis of recycling results in multiple cycles

To investigate the stability of the performance of nanocomposites in recycling cycles, the samples were tested after three recycling cycles. The results showed (Table 1) that the mechanical properties of the samples with nanoparticles had a smaller decrease, such that the hardness and tensile stress decreased by only 10% at the end of the third cycle; while in the samples without nanoparticles, this decrease was observed up to 25%.

Table 1
Comparison of key properties for recycled polyethylene

$CaCO_3$ (w %)	Tensile strength (MPa)	Young's modulus (MPa)	Hardness (Shore D)	Thermal Expansion (μm/m°C)	MFR (g/10min)	OIT
0	18.5	900	62	145	9.1	18.02
2	21.7	1100	67	130	8.2	22.04
5	24.2	1250	70	115	7.6	27.01
10	20.1	1150	68	120	7.9	24.50

CONCLUSIONS

The aim of this research was to investigate the effect of adding calcium carbonate nanoparticles to recycled polymer composites based on heavy polyethylene and polypropylene, to improve mechanical, thermal and rheological properties. Results showed that 5 wt% $CaCO_3$ yielded optimal performance, increasing tensile strength and hardness, improving thermal stability, reducing thermal expansion, and maintaining favorable melt flow properties.Utilizing this nanocomposite in multiple recycling cycles will reduce economic costs and reduce polymer waste. Its appropriate combination with bio-based materials also paves the way for the development of sustainable materials. Given the increasing price of polymer raw materials, the use of additives such as calcium carbonate nanoparticle can reduce the final cost of the product. Although the initial cost of nanoparticles may be high, they are considered cost-effective due to improved mechanical properties and increased durability, in terms of product life cycle and reduced failure rates. Also, the ability to restore the properties of recycled composites through nanoparticles will reduce material waste and improve environmental sustainability.

Authors Contribution statement

Hassan Fathinejad conceived and designed the study, collected and analyzed the data, and drafted the manuscript. Roya Sadat Heyderi critically revised the manuscript for important intellectual content. Both authors have read and approved the final version of the manuscript and agree to be accountable for all aspects of the work, ensuring accuracy and integrity.

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