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Review Article

Reduction of Metal Oxides by Pyrolysis of Waste Polymers

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Abstract: This review evaluates the effectiveness of gaseous products derived from the pyrolysis of waste polymers (polyethylene (PE), polypropylene (PP), high-density polyethylene (HDPE) and waste tires) in reducing metal oxides (cobalt(III) oxide (Co_3O_4), nickel(II) oxide (NiO), bismuth(III) oxide (Bi_2O_3) and copper(II) oxide (CuO)). Owing to the high energy consumption and environmental impact of conventional reduction techniques, utilizing pyrolytic gases as an alternative reducing atmosphere is critical for sustainability and resource recovery. Experimental studies conducted using horizontal tube furnace systems have demonstrated that gas mixtures obtained from waste polymers — comprising mainly H_2, CH_4, C_2H_4 and aromatic hydrocarbons — can effectively reduce these metal oxides at relatively low temperatures. This review comparatively evaluates parameters influencing reduction efficiency, such as temperature, reactant ratio, heating rate, and gas composition, and provides detailed evaluations of product phases and microstructures through XRD, SEM, and EDS analyses. The findings reveal novel opportunities for waste management and sustainable metallurgy, establishing pyrolytic gases as a promising solution at scientific and industrial levels.

Keywords: *Metal Oxide, Pyrolytic Gas, Reactive Atmosphere, Reduction Process, Sustainable Metallurgy, Waste Polymer.*

Atık Polimerlerin Pirolizi ile Metal Oksitlerin İndirgenmesi

Öz. Bu inceleme, atık polimerlerin (polietilen (PE), polipropilen (PP), yüksek yoğunluklu polietilen (HDPE) ve atık lastikler) pirolizinden elde edilen gaz halindeki ürünlerin metal oksitleri (kobalt(III) oksit (Co_3O_4), nikel(II) oksit (NiO), bizmut(III) oksit (Bi_2O_3) ve bakır(II) oksit (CuO)) indirgenme etkinliğini değerlendirmektedir. Geleneksel indirgeme tekniklerinin yüksek enerji tüketimi ve çevresel etkisi nedeniyle, pirolitik gazların alternatif indirgeme atmosferi olarak kullanılması, sürdürülebilirlik ve kaynak geri kazanımı için kritik öneme sahiptir. Yatay tüp fırın sistemleri kullanılarak yapılan deneysel çalışmalar, H_2, CH_4, C_2H_4 ve aromatik hidrokarbonlardan oluşan atık polimerlerden elde edilen gaz karışımlarının, bu metal oksitleri nispeten düşük sıcaklıklarda etkili bir şekilde indirgenebildiğini göstermiştir. Bu derleme, sıcaklık, reaktan oranı, ısıtma hızı ve gaz bileşimi gibi indirgeme verimliliğini etkileyen parametreleri karşılaştırmalı olarak incelemekte ve XRD, SEM ve EDS analizleri yoluyla ürün fazları ve mikro yapılar hakkında ayrıntılı değerlendirmeler sunmaktadır. Bulgular, atık yönetimi ve sürdürülebilir metalurji için yeni fırsatlar ortaya koyarak, pirolitik gazları bilimsel ve endüstriyel düzeyde umut verici bir çözüm olarak ortaya koymaktadır.

Anahtar Kelimeler: *Metal Oksit, Pirolitik Gaz, Reaktif Atmosfer, İndirgeme Prosesi, Sürdürülebilir Metalurji, Atık Polimer.*

1. Introduction

Global plastic production now exceeds 460 million tons per year, and approximately 20 million tons of plastic litter enter the environment annually; yet only a small proportion of this waste is recycled or converted into energy [1]. Notably, as of

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2019, only about 9% of total plastic waste was recycled and 19% was incinerated with energy recovery [2]. A significant proportion of single-use plastics and packaging materials end up in landfills or are released directly into the environment. Inadequate recycling infrastructure exacerbates plastic pollution and hinders sustainable waste management. Against this backdrop, the pyrolysis of waste polymers is attracting attention due to its potential to mitigate environmental impacts, facilitate resource recovery, and foster the development of alternative value chains. Pyrolysis is a thermochemical recycling process that takes place in an oxygen-free environment. This enables plastic waste to be transformed into gaseous, liquid, and solid products [3,4]. These products can be utilized in energy production, chemical synthesis, and metallurgical processes, thereby contributing to the mitigation of plastic waste and the advancement of the circular economy.

Polyethylene (PE), polypropylene (PP), polycarbonate (PC) and waste tires are high-volume polymer types that are widely dispersed in the environment and pose a persistent ecological threat due to their resistance to degradation. When such plastics are disposed of by incineration, toxic gases and residues are generated, introducing additional risks to human health. Consequently, there has been a surge in interest in chemical recycling technologies, particularly pyrolysis-based approaches, in recent years [5].

Transition metals are strategically significant due to their unique properties, such as high melting points, multiple oxidation states, excellent mechanical strength and superior electrical conductivity [6–8]. Elements such as nickel (Ni), cobalt (Co), copper (Cu) and bismuth (Bi) are classified as both transition metals and critical raw materials. Converting their oxide forms back into metallic states generates economic value and enhances resource sustainability, given the limited natural reserves of these elements. Thus, reducing metal oxides is a process of metallurgical efficiency and strategic importance for resource management [9].

Conventional reduction methods include hydrogen reduction, carbon monoxide reduction, and carbothermal processes. However, these approaches require high temperatures, pure reactants, and fossil fuel-based energy. Furthermore, they often present challenges in terms of process control and the generation of unwanted by-products. Therefore, the use of gases derived from the pyrolysis of waste polymers as an alternative reducing atmosphere has gained traction due to its environmental and economic advantages [10, 11]. Pyrolytic gases, which are typically composed of H_2, CH_4, C_2H_2, C_2H_4, and aromatic hydrocarbons, offer strong reduction potential, are low cost and are derived from waste. Their composition can be tailored depending on the type of polymer waste and pyrolysis conditions, enabling optimization of the reduction process for specific oxides.

Horizontal tube furnace systems are commonly used in these processes as they allow the simultaneous and controlled production of pyrolytic gases and the reduction of metal oxides [12]. In these systems, polymer waste is thermally decomposed in an inert atmosphere to generate a gaseous phase, which is then directed towards oxide beds within the same setup to initiate reduction reactions. This two-zone configuration offers advantages in terms of temperature control, gas flow regulation and process monitoring. However, challenges such as limited gas–solid interaction, imbalances in carrier gas flow, and temporal variation in gas composition due to the open system design also arise [13].

Reducing metal oxides using pyrolytic gases has significant advantages over conventional methods, including enabling lower-temperature operations, promoting environmental sustainability through waste reuse, and reducing reliance on fossil-based reductants [14]. Gases derived from the pyrolysis of polymers such as PE, PP, PC and tires contain hydrogen and hydrocarbons with high reducing potential. The composition of these gas mixtures depends on the type of waste, the pyrolysis temperature, and the atmospheric conditions, allowing for optimization tailored to specific applications [15]. In addition to gas composition, the thermal profile and flow dynamics within the reactor are critical factors influencing reduction efficiency. Therefore, a comprehensive approach is necessary to optimize process parameters [16]. Previous studies on the reduction of metal oxides have predominantly focused on conventional reducing agents such as hydrogen or natural gas [17,18]. However, research employing waste-derived reducing media, particularly those generated by polymer pyrolysis, remains limited. Only a few studies have explored the simultaneous reduction and carburization of metal oxides under such conditions, while most have treated these processes separately [19–22]. This review contributes to the literature by systematically integrating recent advances on the use of waste polymer pyrolysis products as reducing agents for metal oxides. The originality of the work lies in its dual focus on thermodynamic mechanisms and sustainability perspectives, providing a comprehensive evaluation that bridges polymer waste valorization with metallurgical reduction and carburization processes.

Within this framework, pyrolysis-based reduction technologies open up new possibilities for metallurgical applications, contributing to the circular economy by enabling the recovery of valuable metals from waste plastics. This review systematically examines the scientific literature on reducing metal oxides (Co_3O_4, NiO, Bi_2O_3 and CuO) using pyrolytic gases. It offers comparative evaluations in terms of polymer types, experimental conditions, reactor configurations, product phases and microstructural properties, providing a comprehensive overview of current knowledge.

2. Experimental Approach

Figure 1 illustrates the schematic representation of the experimental setup employed in reduction studies using waste polymer as a reducing agent source, comprising a horizontal tube furnace with gas inlet systems and a gas flow meter. The system consists of two distinct heating zones. In the first zone, waste polymers such as polyethylene (PE), polypropylene (PP), and waste tires were placed in alumina boats and heated under a high-purity argon atmosphere. Typical pyrolysis temperatures ranged from 550–800 K for plastics, and 800–900 K for tires. During thermal decomposition, the wastes released gaseous species including H_2, CH_4, C_2H_4, and C_2H_2, generating an in-situ reducing atmosphere. Simultaneously, the metal oxides (Co_3O_4, NiO, Bi_2O_3, and CuO) were positioned in the second heating zone, which was maintained between 800 and 1000 K. The pyrolytic gases produced in the first zone were carried by the flowing Ar directly over the oxide bed, enabling gas–solid reduction. Both zones were heated at a ramp rate of 15 K min^{-1} and then held isothermally at the target temperature. The distance between the two alumina boats was approximately 25 mm in the CuO and Bi_2O_3 experiments. For the NiO and Co_3O_4 systems, the same dual-zone horizontal configuration was used, ensuring effective gas contact while preventing solid mixing.

The polymer wastes used as reducing-agent precursors were cut into 2–3 mm pieces prior to the experiments. Thermogravimetric analysis (TGA) of these wastes under argon revealed that the volatile-matter contents were approximately 95–96 wt.% for HDPE and PP and about 66 wt.% for waste tires, the remainder consisting of carbon black ($\approx$ 30 wt.%) and minor ash ($\approx$ 4 wt.%, mainly ZnO and SiO_2).

For each experiment, 0.05–0.09 g of metal oxide was used, while the amount of waste polymer varied according to the selected reactant ratio ($m_{(waste)}/m_{(oxide)}$ = 0–10, depending on the system). Argon flow rate was 42.5 cm^3 min^{-1}, which provided continuous gas transport and prevented external air ingress.

The extent of waste pyrolysis and oxide reduction was determined based on mass change. Sample masses were measured before and after each run using an analytical balance with a precision of $\pm 10^{-4}$ g. The change in mass was expressed as Mass (%) = $(m_p/m_0) \times 100$, where m_0 and m_p represent the initial and final (product) masses, respectively. A decrease in mass directly indicates the progress of waste decomposition and metal-oxide reduction. Although different oxides (NiO, Co_3O_4, CuO, and Bi_2O_3) were investigated, all experiments followed the same in-situ gas–solid reduction methodology.

The reduced products were subsequently characterized by x-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive x-ray spectroscopy (EDS) to determine the phase composition and microstructural features.

3. Reduction of Metal Oxides

This section presents the results of experiments on the reduction of transition metal oxides (Co_3O_4, NiO, Bi_2O_3 and CuO) using pyrolytic gases derived from various waste polymers and describes the phase transformations and reduction mechanisms involved.

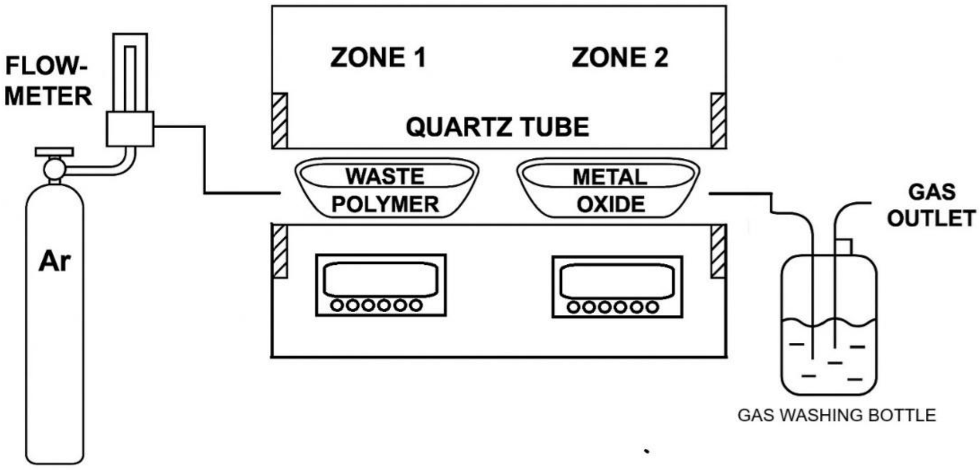

Figure 1. *The schematic representation of the experimental setup employed in the reduction of metal oxide by waste polymer pyrolysis.*

3.1. Reduction of Co_3O_4 Using Waste Tires and HDPE

Cumbul Altay and Eroğlu (2021) investigated the gas-phase species produced from the pyrolysis of waste tires and HDPE for their ability to reduce Co_3O_4. The study elucidated the conditions under which the reduction sequence $Co_3O_4 \rightarrow CoO \rightarrow Co$ proceeds effectively. In these experiments, Co_3O_4 powder and waste tire fragments were placed in separate alumina boats inside a quartz tube furnace and heated to 900 K at a rate of 15 K/min. Around 66% of the tire mass was converted into gas, which was then transported by argon to the oxide bed. Increasing the amount of waste was found to enhance the degree of reduction. However, full conversion to the metallic Co phase only occurred upon the addition of HDPE. Maximum reduction efficiency was reached at an HDPE/tire ratio of 0.1, while higher ratios led to carbon deposition and mass gain. XRD analysis confirmed complete reduction when the sample was held at 900 K for 30 minutes in an HDPE-enriched atmosphere. The diffraction peaks and their intensities matched those of the standard f.c.c. Co reference, confirming full transformation. These phase analysis results were consistent with mass loss measurements [10].

3.2. Reduction of Co_3O_4 Using Waste HDPE

Cumbul Altay and Eroğlu (2022) thoroughly examined the reduction behavior of Co_3O_4 under a gas atmosphere derived from waste polyethylene. The study focused on determining the optimal conditions for the formation of a single phase of cobalt and clarified the reduction mechanism involving pyrolytic gases. PE and Co_3O_4 samples were placed 20 mm apart in alumina boats and heated under 42.5 sccm of argon gas at heating rates of 5 and 15 K/min within the range of 650–

900 K. At a PE-to-Co_3O_4 ratio of 1, nearly complete reduction ($\alpha \approx 0.96$) occurred at 800–900 K; however, limited pyrolysis and reduction were observed at 650 K. Complete reduction was also achieved at 750 K following a 60-minute isothermal hold. XRD confirmed the $Co_3O_4 \rightarrow CoO \rightarrow Co$ pathway. Following isothermal treatment at 750 K, the CoO peaks diminished and the f.c.c. Co peaks became dominant. At 800 K, only metallic Co was observed. SEM images revealed that the initially smooth, octahedral Co_3O_4 particles transformed into porous, sponge-like Co structures due to the effects of gas diffusion and internal pressure during reduction [11].

3.3. Reduction of NiO Using Waste Tires

Cumbul, Altay and Eroğlu (2019) investigated the effectiveness of pyrolytic gases obtained from waste tires in reducing NiO. This study was one of the first to experimentally evaluate gas-phase reduction using tire-derived pyrolysis gases in a horizontal quartz tube furnace system. Two separate alumina boats — one containing waste elastomer and the other containing nanocrystalline NiO (average particle size 12 nm) — were placed 120 mm apart in the furnace. Under an argon atmosphere (42.5 sccm) and with a heating rate of 15 K/min, the system was raised to 1000 K and held isothermally for 60 minutes. The gaseous species generated from the pyrolysis of tire were carried to the NiO bed, resulting in effective reduction. XRD analysis revealed that a reactant mass ratio (m_{tire}/m_{NiO}) of 0.734 produced only metallic Ni, whereas a lower ratio (0.367) produced a mixture of Ni and residual NiO phases. SEM images revealed that lower tire content resulted in relatively large Ni particles (~2 µm) forming, whereas higher content caused carbon to diffuse into the metal surface, forming finer particles (~0.1 µm) due to a metal dusting effect [13].

3.4. Reduction of NiO Using Waste HDPE

Another 2019 study by Cumbul Altay and Eroğlu systematically evaluated the use of HDPE-derived pyrolytic gas for reducing NiO. The high carbon and hydrogen content of HDPE was considered an advantage as a potential gas-phase reducing agent. HDPE and NiO samples were placed in a quartz tube furnace and heated under an argon flow between 550 and 900 K. According to thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) results, HDPE began to pyrolyze at approximately 670 K, completing decomposition at around 900 K. As the pyrolysis temperature increased, both the gas yield from the HDPE and the degree of reduction of the NiO improved. Complete reduction was achieved at 900 K. XRD results revealed the presence of both NiO and metallic Ni at 750 K and only metallic Ni at 900 K. SEM and EDX analyses showed that 100 nm Ni particles formed at 750 K while sintering caused the development of larger particles (approximately 180 nm) at 900 K. This indicates the significant influence of the reduction temperature on the final particle morphology [19].

3.5. Reduction of NiO Using Waste PP

The potential of pyrolytic gases from polypropylene waste in reducing NiO was studied by Cumbul, Altay, and Eroğlu (2019). This research investigated the hypothesis that hydrocarbons produced through the pyrolysis of plastic waste can serve as effective gas-phase reductants. Experiments were conducted at temperatures between 600 and 900 K in a horizontal quartz tube furnace using 0.025 and 0.05 g of polypropylene (PP). PP decomposition began at ~600 K and was almost complete at ~800 K. XRD and SEM–EDX analyses confirmed that only the higher amounts of PP (0.05 g) resulted in the complete reduction of NiO, yielding solely metallic Ni with particle sizes of around 70 nm. In contrast, a limited reduction occurred with 0.025 g of PP. The study also estimated that, due to the open configuration of the system, only ~15% of the generated gases interacted effectively with the oxide. This accounts for the discrepancy between theoretical and experimental reduction efficiencies [20].

3.6. Reduction of Bi_2O_3 Using Waste Tires

Cumbul, Altay and Eroğlu (2019) also evaluated the reduction of Bi_2O_3 using pyrolysis gases derived from waste tires, employing a combination of experimental and thermodynamic methods. Bi_2O_3 is usually reduced at temperatures of at least 1073 K using solid carbon. However, this study demonstrated partial and complete reduction at significantly lower temperatures (700–900 K) via gas-phase species such as hydrogen and hydrocarbons. In the horizontal quartz furnace, waste tires and Bi_2O_3 samples were placed 25 mm apart in alumina boats. With a heating rate of 15 K/min and an argon flow of 42.5 sccm, the system was heated to 900 K. XRD results showed partial reduction to a mixture of Bi, β-Bi_2O_3 and α-Bi_2O_3 phases. However, full reduction was achieved when a HDPE/tire ratio of 0.36 was introduced, yielding only rhombohedral bismuth. SEM analysis revealed that the reduced bismuth formed coalesced metallic droplets ⩾1000 µm in diameter, driven by liquid-phase migration and coalescence initiated at the oxide surface [21].

3.7. Reduction of CuO Using Waste Tires

In a 2021 study, Cumbul Altay and Eroğlu investigated the reduction of CuO via gases obtained from the pyrolysis of waste tires. The study examined the reaction behavior at temperatures between 600 and 900 K across various tire-to-CuO ratios, as well as the impact of HDPE additives. The CuO and tire pieces were placed 25 mm apart and heated at a rate of 15 K/min in argon. Complete reduction was achieved at 900 K for a tire/CuO reactant ratio of 1.28, which aligns with thermodynamic predictions. At higher ratios (e.g. 8.8), however, mass gain was observed due to carbon deposition on the Cu surfaces. Co-pyrolysis experiments with HDPE revealed an enhanced reduction efficiency, with full reduction occurring at both 800 and 900 K at an HDPE/tire ratio of 0.35. XRD confirmed the exclusive presence of metallic Cu. SEM images showed that the initial flower-like morphology of the CuO transformed into equiaxed particles due to surface-initiated reduction, structural cracking and sintering [22].

4. Comparative Summary of Process Parameters and Results

To provide a concise comparison of all experimental systems investigated, Table 1 summarizes the key process parameters and main outcomes. The table includes the reference number, reaction temperature (K), waste-to-oxide mass ratio, mass % (($m_{product}/m_{oxide}$) × 100), XRD findings, and SEM observations. This comparative overview highlights the differences in reaction behavior among the systems and complements the detailed discussions presented in Sections 3.1–3.7.

Table 1. Comparative summary of process parameters and results for different waste–metal oxide systems under Ar flow (42.5 cm^3min^{-1})

Ref.	**Temp. (K)**	**Waste/oxide mass ratio**	**Mass (%)**	**XRD findings**	**SEM observations**
[10]	900	m_{tire}/m_{Co3O4} =14.08 m_{HDPE}/m_{tire} =0.1	73.5%	$Co_3O_4 \rightarrow CoO \rightarrow Co$ (+ minor C)	Pothole morphology from Co_3O_4–Co phase densification
[11]	800	m_{HDPE}/m_{Co3O4} = 1	73.5%	$Co_3O_4 \rightarrow CoO \rightarrow Co$	Spongy Co particles
[13]	1000	m_{tire}/m_{NiO} =0.734	78.6%	NiO → Ni	Coarse Ni particles (sizes>2 μm)
[19]	900	m_{HDPE}/m_{NiO} =0.56	78.57 %	NiO → Ni	Fine Ni particles (~ 180 nm)
[20]	800	m_{PP}/m_{NiO} =0.56	78.5%	NiO → Ni	Ultrafine Ni particles (~ 70 nm)
[21]	900	m_{tire}/m_{Bi2O3} =10 m_{HDPE}/m_{tire} =0.36	89.6%	$Bi_2O_3 \rightarrow Bi$	Large Bi spheres (shots)
[22]	900	m_{tire}/m_{CuO} =1.28 m_{HDPE}/m_{tire} =0.35	82.8% 81.2%	$CuO \rightarrow Cu_2O \rightarrow Cu$	Flower type CuO→ equiaxed Cu particles

5. Conclusion

This review has comprehensively addressed the role of pyrolytic gases derived from waste polymers in reducing transition metal oxides. Literature reviews have demonstrated that oxides such as Co_3O_4, NiO, Bi_2O_3 and CuO can be effectively reduced at low temperatures using gases generated through the pyrolysis of PE, PP, HDPE and waste tires. The high reduction potential of pyrolytic gases, which are primarily composed of H_2, CH_4, C_2H_4 and aromatic hydrocarbons, makes this method environmentally favorable and economically viable.

Experimental findings confirm that parameters such as pyrolysis temperature, reactant ratio, heating rate and isothermal holding time directly influence the resulting product phases and microstructures. Supported by XRD and SEM analyses, the reduction pathways typically follow a sequence from the oxide phase to the intermediate phase and finally to the metallic state, with high-purity metallic products being obtained under optimized conditions.

Using pyrolytic gases from waste polymers for oxide reduction is not only a promising metallurgical method, but also significantly increases environmental sustainability. This approach promotes the valorization of plastic waste as an alternative to harmful disposal methods while reducing reliance on fossil-derived reducing gases. Consequently, it contributes to reducing greenhouse gas emissions and supports the development of circular, low-carbon metallurgical processes. Furthermore, this method offers a viable alternative for metal recovery from secondary resources.

In future studies, emphasis should be placed on optimizing gas composition, advancing continuous-flow systems and evaluating the performance of the reduced products. In this context, pyrolysis-based reduction technologies emerge as innovative, applicable tools that align with the goals of sustainable metallurgy.

Author Contribution

Formal analysis – (MCA); Investigation – MCA (SE); Experimental Performance – MCA; Collection – SE; Processing – MCA; Literature review – MCA and SE; Writing – MCA; Review and editing – SE.

Declaration of Competing Interest

The authors declared no conflicts of interest with respect to the research, authorship, and/or publication of this article.

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