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Research on the Reverse Flotation of Waste Printed Circuit Boards

Theses booklet of Doctoral (PhD) dissertation

by

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1. Background

The rapid proliferation of consumer electronics has led to an unprecedented rise in electronic waste (e-waste), now one of the world's fastest-growing solid waste streams [1]. In 2022 alone, global e-waste generation reached 62 million metric tonnes, with only a fraction formally recycled [2]. This uncontrolled growth poses both a challenge and an opportunity: while e-waste contains hazardous materials that threaten health and the environment, it also serves as an urban mine rich in base and precious metals. Efficient recycling is therefore imperative, not only to reduce environmental risks and resource depletion but also to support circular economy targets through material recovery and energy conservation [3].

Among e-waste fractions, waste printed circuit boards (WPCBs) stand out due to their high metallic content and complex material composition. Though they comprise just 3–7 wt.% of e-waste, WPCBs contain up to 30–50 wt.% metals, including over 20% copper and valuable quantities of gold, silver, and palladium [4, 5]. Economically, their recycling potential is significant; for example, a small-scale hydrometallurgical plant processing 500 tonnes of high-grade WPCBs over ten years was estimated to yield a net present value of up to €63 million under average market conditions [6]. However, their heterogeneity, encompassing metals, thermoset resins, fiberglass, and ceramics, renders them difficult to process using conventional techniques.

Current WPCB recycling practices typically involve multi-stage flowsheets, including manual disassembly, mechanical pretreatment, and downstream metallurgical recovery via pyrometallurgy or hydrometallurgy [7]. While direct metallurgical processing is feasible, it is inefficient and environmentally burdensome without adequate preprocessing [8]. Mechanical pretreatment is essential to liberate metallic components and concentrate valuable fractions, yet it also generates fine particles that reduce separation efficiency and exacerbate metal losses [9]. To address these limitations, froth flotation has emerged as a promising separation technique, particularly for concentrating fine metallic fractions while maintaining low energy demand (Figure 1).

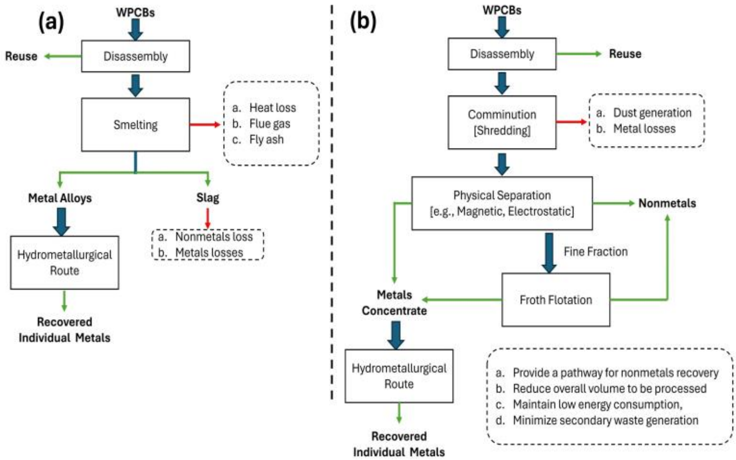

Figure 1: Simplified process flowsheets illustrating (a) direct metallurgical approach and (b) incorporating mechanical preprocessing prior to metallurgical processing (adapted from Das et al. [9]).

Froth flotation, long established in mineral processing, is a three-phase separation technique that exploits differences in surface hydrophobicity [10]. In WPCB systems, reverse flotation is preferred: hydrophobic nonmetallic materials are floated and rejected, enriching metallic particles in the underflow. The feasibility of this approach has been demonstrated at the laboratory scale, leveraging the natural hydrophobicity of nonmetals for selective separation [11]. However, despite growing academic interest and technical potential, the flotation of WPCBs has yet to find industrial applications. Its scalability is limited by interrelated challenges involving feed preparation, pulp particle dispersion, flotation hydrodynamics, solids loading, metal-specific flotation behavior, and pulp rheology. These challenges stem from the unique physical and chemical properties of WPCBs, which must be addressed holistically to enable scalable, high-efficiency processing.

2. Research Aim and Objectives

The aim of this PhD dissertation is to provide a comprehensive understanding of the key factors governing the reverse flotation of WPCBs, as well as to propose scientific and practical strategies for scalable and efficient processing.

To achieve this aim, the dissertation explores the influence of grinding environments on flotation feed preparation; identifies the aggregation and dispersion behaviors of WPCB particles in aqueous suspension and the role of intense agitation; examines the impact of flotation hydrodynamics and solids concentration on metal recovery dynamics; characterizes pulp rheology and its effect on flotation performance; and evaluates the effectiveness of ultrafine removal and alkaline-assisted grinding as pretreatment strategies.

3. Experimental

Materials

A representative batch of mixed populated and unpopulated waste printed circuit boards (WPCBs) was sourced from an e-waste facility in Budapest. The sample included multilayer, double-sided, and single-sided boards from various electronic devices such as computers, servers, and household appliances. A subset of high-layer-count FR-4 boards was separately identified. No hazardous components (e.g., batteries or displays) were present. Key chemicals used in flotation and auxiliary procedures included Aerofroth 70 (alcohol- and glycol-based) as the frother, sodium hydroxide and hydrochloric acid for pH control, n-heptane for liquid-liquid separation tests, and ethanol for density measurements.

Methods

The experimental investigations were conducted using tailored methodologies to address specific factors governing the reverse flotation of WPCBs. The experimental work is structured into four main sections as follows:

I. Mechanical processing and grinding

Mechanical processing of WPCBs was carried out through a staged comminution and separation approach aimed at achieving selective liberation and material upgrading prior to flotation. A combination of axial gap rotary shear, hammer shredder, and high-speed impact milling was applied to reduce board size and liberate metals. This was followed by multiple physical separation steps, including magnetic, eddy current, and electrostatic methods, to recover liberated metals at a coarser size. The post-separation fraction, representative of industrial rejects, was then subjected to controlled wet and dry grinding using a laboratory tumbling ball mill to adjust particle size distribution (PSD) and enhance liberation. Characterization focused on particle size (via sieve analysis and dynamic image analysis), liberation degree (via optical and SEM–EDS imaging), metallic particle shape (via optical microscopy and ImageJ), and chemical composition (via ICP-OES).

II. Aggregation/Dispersion challenges and the role of intense agitation

To investigate the impact of particle aggregation and dispersion, a series of flotation experiments were conducted using a 1 L mechanical flotation cell, following a standardized reagent regime. Prior to flotation, samples underwent intense agitation in a baffled tank at stirring speeds ranging from 500 to 2000 rpm for 15 minutes. The influence of agitation on dispersion was evaluated through post-conditioning wet sieving, which led to the proposal of an aggregation index (AI) by comparing wet and dry PSDs. Zeta potential measurements were also performed to assess surface charge variations across different conditioning strategies. Final flotation performance was quantified via separation efficiency (S.E.), derived from metal grades in the feed, concentrate, and tailings.

III. Flotation Performance and Pulp Properties

To evaluate flotation performance under varying cell variables, WPCB feed pretreated by intense agitation was subjected to reverse flotation in a 1 L mechanical cell. A Box-Behnken design was applied to assess the effect of impeller speed, air flow rate, and solids concentration on flotation performance. Key diagnostics included pulp rheology (measured via vane-in-cup rheometry), water recovery (determined gravimetrically), flotation kinetics (modeled using four established kinetic equations), and entrainment (quantified via Ross's method). Complementary two-liquid phase separation tests using a water–n-heptane system were conducted to assess particle hydrophobicity. These combined evaluations provided a detailed understanding of how flotation variables and pulp properties govern both metal and nonmetal flotation behaviors in WPCB systems.

IV. Mitigating Rheological Challenges in the Flotation of WPCB

To address rheological limitations at elevated solids concentrations, two pretreatment strategies were investigated: physical removal of ultrafines and chemical modification via alkaline grinding. The physical approach involved screening out particles <0.032 mm to reduce ultrafine-induced viscosity buildup. The chemical strategy integrated sodium hydroxide into wet and dry ball milling, modifying pulp chemistry and surface properties. Flotation tests were conducted under fixed hydrodynamic conditions to evaluate performance, with rheological behavior measured for each pretreatment scenario. FTIR spectroscopy was used to assess chemical changes in nonmetallic phases post-grinding, while potential metal leaching during alkaline treatment was evaluated using AAS for the presence of metal ions. Together, these methods enabled assessment of how physical and chemical modifications impact flotation efficiency and metal recovery under high-solid, viscosity-sensitive conditions.

4. New Scientific Findings, Claims

Claim 1: It was established that the inherent hydrophobicity of WPCB nonmetallic particles governs the processing behavior of post-separation fractions from grinding through to flotation, where hydrophobic particle interactions influence dispersion, rheology, and ultimately flotation performance. The optimal processing approach is outlined as follows:

- A staged processing strategy of dry environment grinding followed by intense wet agitation effectively limits aggregation during comminution and enhances dispersion during flotation pulping.
- Hydrophobicity control through chemical modification enables rheological improvement, allowing operation at higher solids concentrations.

Claim 2: It was revealed that hydrophobic interactions among nonmetallic WPCB particles during wet ball milling create a rheology-dependent stress mechanism that reduces breakage efficiency

compared to dry grinding, where superior particle dispersion enables more effective size reduction of WPCB post-separation fractions.

Claim 3: It was proven that the formation of a stabilized foam layer at the pulp–air interface under high turbulence conditions represents a distinct hydrophobicity-induced pathway of metallic losses during flotation, negatively influencing WPCB flotation performance.

- This foam layer (Figure 2) was experimentally proven to arise from turbulence-driven air entrainment, subsequently stabilized by fine hydrophobic nonmetallic particles (<0.075 mm). This layer acts as a physical barrier that entraps metallic particles, leading to their direct transport to the overflow upon the initiation of the flotation process.
- Direct foam layer assays confirmed substantial metal accumulation, with 5.7 g/kg of copper and 22.6 mg/kg of gold. Corroborating this, overall flotation results demonstrated that metallic losses exceeded 45% following high stirring speed agitation.

Figure 2: Foam layer at the pulp-air interface (no aeration or frother addition).

Claim 4: It was confirmed that energy input (stirring speed) and duration during intense agitation pretreatment govern the dispersion state of WPCB pulps by modulating hydrophobicity-driven aggregation and foam formation, thereby influencing flotation performance.

- Suboptimal agitation energy led to the persistence of hydrophobic particle aggregation, while optimal energy input achieved maximum dispersion efficiency. Exceeding optimal energy levels caused detrimental foam layer formation, with flotation performance confirming the critical dispersion-flotation relationship (Figures 3 and 4).

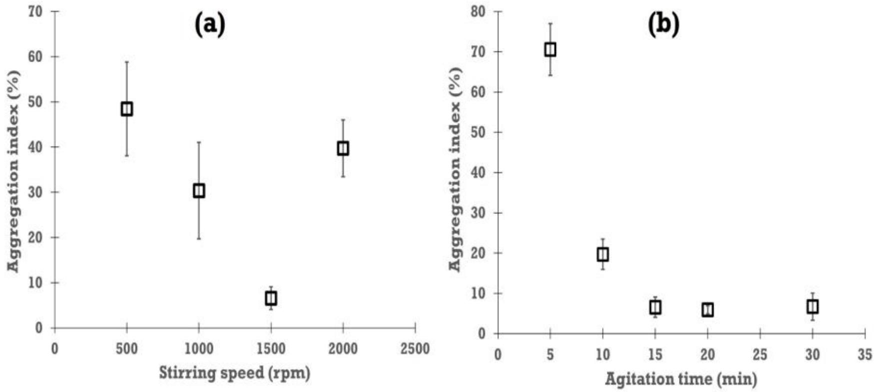

Figure 3: Effect of (a) agitation energy and (b) duration on the aggregation index during the intense agitation pretreatment of WPCB.

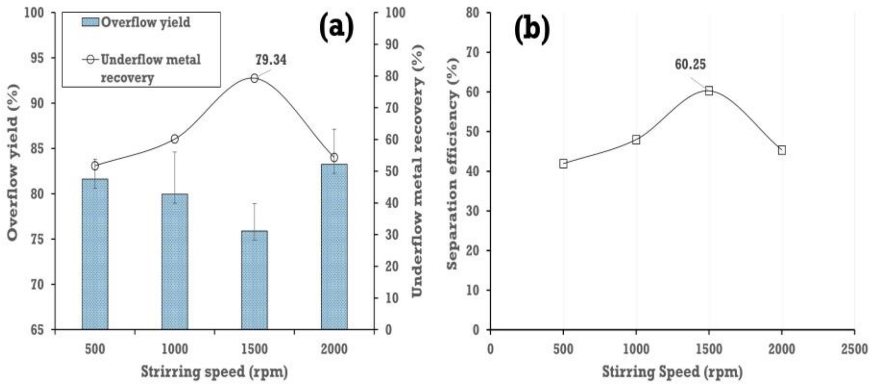

Figure 4: Effect of stirring speed during intense agitation pretreatment on flotation performance: (a) overflow yield and underflow metal recovery, and (b) separation efficiency.

Claim 5: It was revealed that copper and gold, representing the dominant bulk and value metal targets, respectively, exhibit distinct and competing recovery trends under varying pulp solids concentrations in a single-stage reverse flotation, demanding trade-off optimization.

- Box-Behnken analysis revealed divergent responses (Figure 5).
- Response surface numerical optimization resulted in three scenarios (Figure 6).

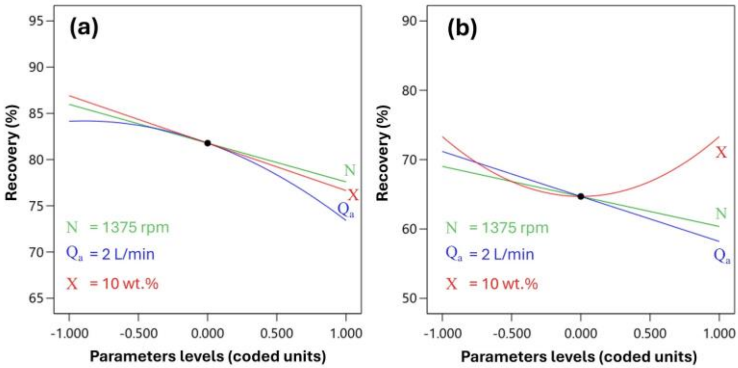

Figure 5: Main effects of variables on (a) copper recovery and (b) gold recovery.

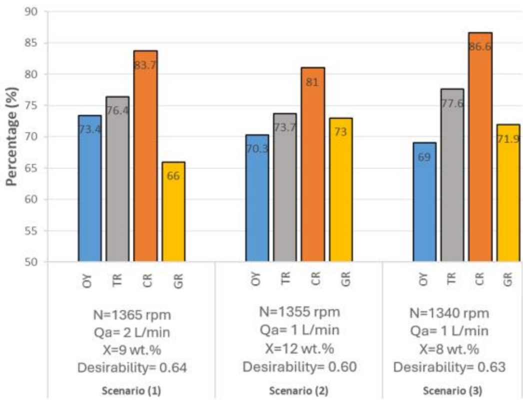

Figure 6: Flotation outcomes across optimization scenarios (OY: overflow yield, TR: total recovery, CR: copper recovery, GR: gold recovery).

Claim 6: It was proven that the low-solids concentration constraint in WPCB flotation is governed by rheological limitations, where severe apparent viscosity buildup disrupts key flotation mechanisms.

- Apparent viscosity increased sharply from 1.67 to 23.91 mPa·s as solids concentration rose from 6 to 18 wt.% (Figure 7).
- Rheological behavior is primarily caused by hydrophobic interparticle attraction and secondarily by frictional effects of elongated nonmetallic particles.
- Flotation performance declined significantly beyond 10 wt.% solids due to viscosity-induced disruptions in gas dispersion, aggregate stability, and froth mobility.

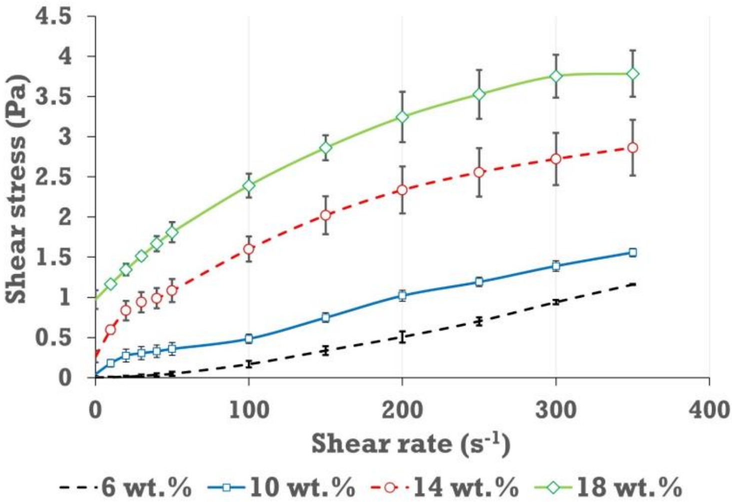

Figure 7: Rheological behavior of WPCB flotation feed suspension at 6, 10, 14, and 18 wt.% solids.

Claim 7: It was revealed that metallic particle losses to the overflow in WPCB reverse flotation are governed by a mass-pull mechanical entrapment mechanism. For coarse and intermediate-sized metals (0.075-0.250 mm), losses are driven by viscosity-enhanced mechanical entrapment under high-solids conditions; for fine-sized metals (<0.075

mm), losses arise from viscosity-independent mechanical entrapment, mobilized by bulk-phase upward transport.

- For coarse (0.125–0.250 mm) and intermediate (0.075–0.125 mm) metals, flotation at 14 wt.% solids resulted in substantial overflow losses (41.8% and 18.35%, respectively), despite high liberation (85–90%) and unfavorable conditions for true flotation. Rapid loss kinetics (~60% within 1 minute) and visual recovery of dense, non-floatable metals in the froth confirm that elevated pulp viscosity suspended these particles, reducing drainage and promoting mechanical entrapment.
- For fine metals (<0.075 mm), overflow losses remained high across all pulp conditions (14.09% at 6 wt.% to 59.72% at 14 wt.%). High entrainment indices (1.9–8), faster kinetics than water recovery, and Kelsall slow-fraction contributions below 37% indicate that losses were driven by viscosity-independent mechanical entrapment, a bulk-phase transport mechanism in which fine particles are swept upward within the pulp.

Claim 8: It was established that the removal of ultrafine particles (<0.032 mm) from high-solids WPCB flotation feed (14 wt.%) effectively mitigates rheological limitations, enhancing metal recovery, but introduces trade-offs by compromising froth stability and nonmetal rejection.

- Ultrafine removal reduced the pulp's yield stress from 0.35 Pa to 0.10 Pa and apparent viscosity (at 100 s^{-1}) from 16.0 to 5.13 mPa·s, restoring near-Newtonian behavior (Figure 8).
- Rheological improvement translated to metallurgical gains: underflow metal recovery increased across all size fractions, with overall metal recovery rising from 68.48% to 83.64% and separation efficiency from 42.66% to 54.78% (Figure 9).
- The modified pulp exhibited reduced froth volume and larger bubble sizes despite similar viscosity to an unmodified 10 wt.% pulp, indicating that interface-active ultrafines contribute to froth stability. Their absence weakened froth structure, limiting nonmetal rejection.

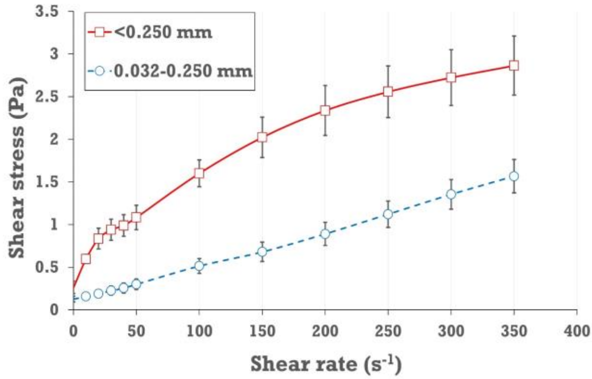

Figure 8: Rheology of original vs. modified feed (14 wt.% solids).

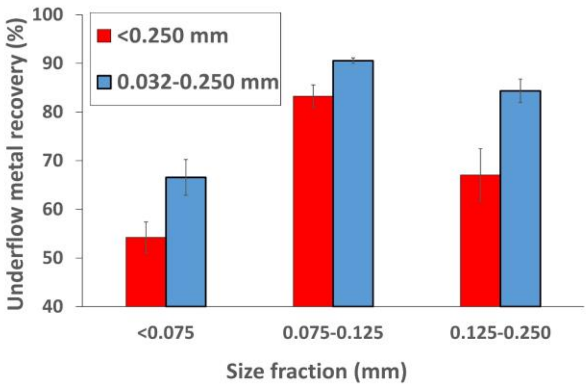

Figure 9: Underflow metal recovery per size fraction.

Claim 9: It was established that alkaline conditions introduced during wet grinding chemically modified WPCB nonmetallic surfaces via hydroxylation, promoting nonmetal dispersion, reducing particle aggregation, and enhancing grinding efficiency.

- FTIR analysis confirmed functional group transformation during wet alkaline grinding (Figure 10).
- Zeta potential shifted from –30.00 mV (standard) to –39.91 mV (wet alkaline pH 12), indicating increased surface hydroxylation.

- pH adjustment from neutral to 10 reduced X_{80} from 0.36 mm to 0.33 mm and increased sub-0.250 mm fraction from 61.90% to 67.16%.
- The performance gain is attributed to reduced interparticle hydrophobic attraction and improved dispersion under alkaline conditions, which reduced the tendency toward particle-bed stressing during grinding.

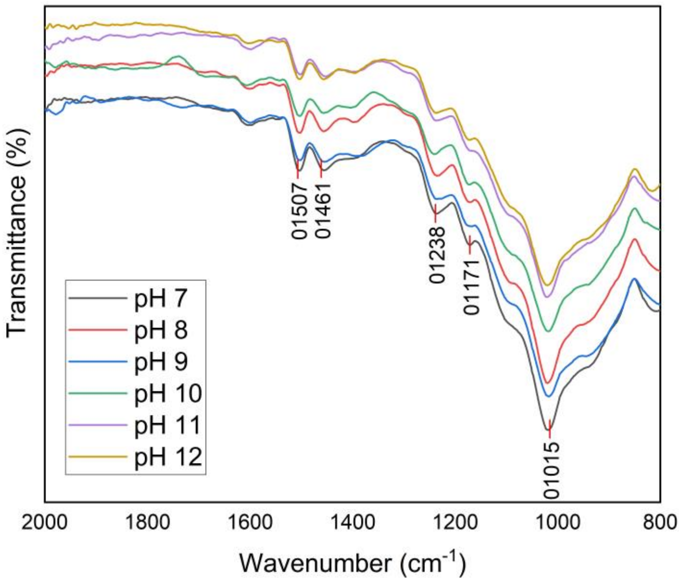

Figure 10: FTIR spectra of WPCBS nonmetals wet grinding under alkaline conditions.

Claim 10: It was proved that grinding under alkaline conditions (0.1 wt.% NaOH) enhances WPCB flotation at an elevated solids concentration of 14 wt.% by modifying pulp rheology and cleaning metallic surfaces.

- The overflow yield and metal recovery peaked at 72.71% and 82.88%, respectively, with 0.1 wt.% NaOH grinding (GD2); higher NaOH dosages resulted in loss of selectivity (Figure 11).
- Rheological analysis showed elimination of yield stress (from 0.35 Pa to 0 Pa) and a 58% reduction in apparent viscosity (from 16 to 6.75 mPa·s) (Figure 12).

- GD2-treated pulp at 14 wt.% solids outperformed untreated pulp at 10 wt.% solids, achieving 82% metal recovery compared to 72%. This improvement, despite similar viscosity, suggests that surface cleaning of metal particles provides additional flotation benefits.

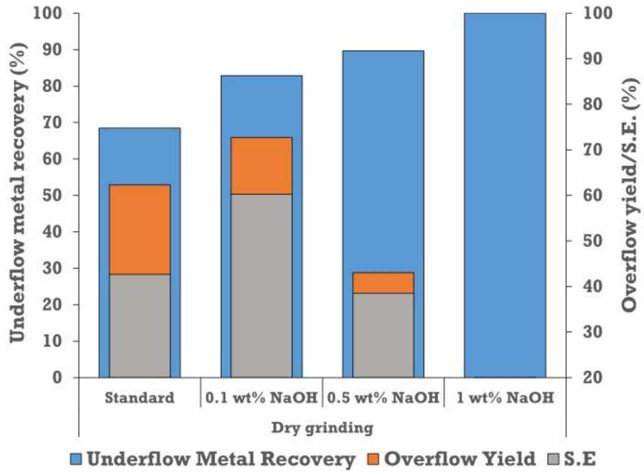

Figure 11: Effect of dry alkaline-assisted grinding on reverse flotation performance (14 wt.% solids).

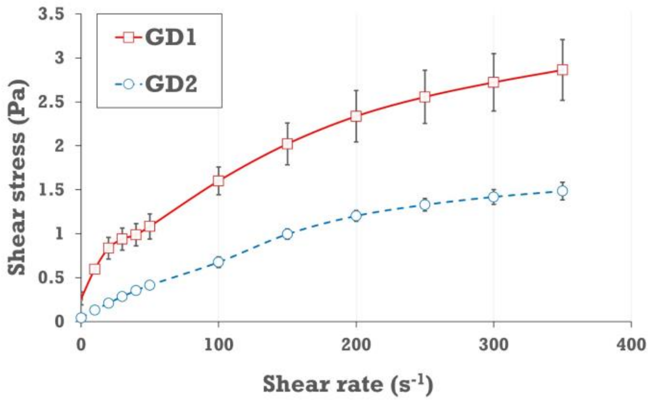

Figure 12: Rheology of standard grinding (GD1) vs. alkaline grinding at 0.1% NaOH (GD2).

Claim 11: It was established that alkaline grinding with 1 wt.% NaOH induces chemical transformation of the WPCB resin matrix, causing ether bond cleavage and surface hydroxylation. This process shifts nonmetal surfaces to a strongly hydrophilic state, effectively depressing them in flotation.

- This outcome creates opportunities for direct flotation schemes, where metals can be selectively floated if made hydrophobic with appropriate collectors, while the depressed nonmetals remain in the underflow, leading to a simplified separation strategy.

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6. List of publications

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