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NEW GENERATION OF ECOFRIENDLY REAGENTS BASED ON ORGANOSOLV LIGNIN NANOPARTICLES: ENVIRONMENTAL ASSESSMENT FOR AN AU FLOTATION CIRCUIT

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Abstract

Froth flotation is the leading process for the selective separation of minerals and the beneficiation of ores. The principle of the method depends on the different wettability properties of the minerals, specifically on the hydrophobicity of the surfaces, which is either inherent or imparted to the minerals through reagents. Despite their widespread and long-standing use, they are considered highly hazardous and toxic and their decomposition poses a potential risk in terms of safety, health, and environmental impact. A new viable alternative, that promotes sustainable development, is the production and utilization of organosolv lignin nanoparticles. This study examines the environmental benefits of producing of lignin nanoparticles from birch wood as and partially replacing xanthate collectors with lignin nanoparticles for the treatment of 1 tonne of mined ore, subjected to flotation. In order to quantify the environmental impact, a life cycle assessment (LCA) was conducted for an Au flotation circuit. The analysis demonstrated that the introduction of organosolv lignin into the flotation process along with the reduction of sodium isopropyl xanthate (SIPX) resulted in the decrease of the environmental footprint and in particular the reduction of climate change and fossil fuel depletion by 16.79% and 3.8%, respectively.

Key words: *flotation, xanthate, lignin, life cycle assessment.*

1. Introduction

1.1. The flotation process

In the mining industry, flotation is a physicochemical method of mineral beneficiation, through which selective concentration and separation from each other or from the gangue is achieved [1].

During the flotation process, the ground ore is placed in the flotation cell where it mixes with water to form a slurry. By introducing air into the cell, the hydrophobic surfaces of the particles adhere to the air bubbles. The density of the bubble-particle system is lower than that of the slurry, causing the rising of the particles to the surface of the flotation cell, and the formation of a froth collected by skimming. The removal of particles from the collected bubbles is achieved through water ionization. Upon the completion of flotation, the froth undergoes thickening and dewatering to minimize the moisture content. The hydrophilic particles remaining in the flotation cell are classified as tailings [2, 3].

The presence of reagents can enhance the efficiency of the flotation process, allowing to obtain products of different desired compositions. One can distinguish reagents into collectors, frothers and modifiers. The selection of the appropriate reagent or the appropriate combination of reagents is a crucial step during the preparation of the flotation method and depends on the process assumption and the ore characteristics [4].

Collectors are organic chemical compounds that are characterized by amphiphilic properties, meaning they contain both hydrophobic and hydrophilic groups. The mechanism of a collector lies in imparting its hydrophobic characteristic to the mineral by adsorbing on its surface. The most frequently used collectors, especially in the flotation of nonferrous minerals are xanthates. Their choice is justified by their high

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efficiency (high selectivity of minerals) and their economic advantage (relatively low cost) [5].

However, the use of xanthates is approached with more concern and it's subject to increasing criticism due to their toxicity. Xanthates can be released into the environment in different ways from manufacturing (e.g. residue generation), transport (e.g. accidental spillage), storage, application and waste disposal (e.g. spillage from litter) [6]. During the hydrolyzation of non-aqueous solutions and flotation pulps, they degrade easily forming toxic substances such as carbon disulfide (CS_2), carbonyl sulphide (COS), alcohol (R-OH), carbonic anion ($2CO^{3-}$) and hydrogen sulphide (H_2S). These compounds pose a serious threat for the environment and humans' safety [7, 8].

1.2. Lignin

Lignocellulosic biomass is the most common organic polymer, accounting for about 1.5 trillion tonnes of total annual biomass production. Its main sources are forest residues and the pulp industry, representing the largest renewable organic source and an inexhaustible supply of raw materials. It is composed of three main polymers: cellulose, hemicellulose, and lignin, which provide lignocellulose with a complex and heterogeneous structure. There is a great variation of the composition and concentration of each polymer according to the type of lignocellulosic biomass. The function of lignin represents 10-25% of the biomass and its chemical structure varies significantly depending on its source [9, 10]. It is a heterogeneous, aromatic, long-chain polymer made up of propanophenol units, mostly connected by ether linkages. Lignin's covalent association with hemicellulose and cellulose molecules hinders the penetration of enzymes that hydrolyse polysaccharides and the extraction of carbohydrates in aqueous solvents, resulting in strong hydrophobic behaviour. Despite containing hydrophilic groups, its three-dimensional structure imparts hydrophobicity and insolubility in water at acidic or neutral pH, though it can dissolve in various organic solvents like ethanol, acetone, and chloroform, as well as in alkaline solutions at high temperatures of 150-180 °C [11].

The three most common types of lignin that can be produced by using different treatment methods are sulfonic lignin, Kraft lignin, and organosolv lignin. The organosolv pre-treatment is particularly effective for biomass delignification, producing clean streams of cellulose, hemicellulose, and lignin. Organosolv lignin (OL) is currently gaining popularity due to its high quality, low ash content, and lack of sulphur compared to Kraft and sulfonated lignin [12].

This study introduces the use of modified flotation reagents with the addition of OL, an economically and ecologically beneficial approach, which promotes European policy and offers a viable solution for the production of renewable, low carbon footprint and economically sustainable collectors. Developing a reagent for the flotation process that uses biodegradable and eco-friendly organosolv lignin as the primary component could decrease the reliance on xanthates and reduce the associated negative impacts, offering a valuable alternative.

2. Experimental

2.1. Purpose and scope definition

Standardized by ISO 14040:2006 and ISO 14044:2006 and guided by the International Life Cycle Data (ILCD) Handbook, life cycle assessment (LCA) evaluates the inputs, outputs of a product or system and the environmental impacts associated with its life cycle. As a methodological framework, it consists of four main phases:

1) goal and scope definition,
2) inventory analysis,
3) impact assessment and
4) interpretation [13].

In the current study, LCA is performed in order to quantify and estimate the environmental impact for the evaluation of the replacement of isopropyl xanthate (SIPX) with a new organosolv lignin-based collector in the flotation process. The scope of the study focuses on examining the environmental advantages of partially replacing xanthate reagents with organosolv lignin nanoparticles (OLN).

Definition of functional unit

The functional unit is defined as 1 kilogram of sulphide ore which is processed to obtain a sphalerite concentrate. The following feed treatment consists mainly of pyrite, arsenopyrite, and the waste from the previous process. The treatment of the ores results in the recovery of pyrite/arsenopyrite concentrate from which gold is extracted as the final product.

System boundaries

For the quantification and evaluation of environmental impacts, a gate-to-gate LCA is conducted and the system boundaries are defined. For the production of the reagent the pretreatment of birch wood biomass to produce organosolv lignin and the production of lignin organosolv nanoparticles are included in the methods boundaries.

Two scenarios were applied for the flotation process. In the base case scenario (BSC), isopropyl xanthate is selected while in the alternative scenario 1 (S1), a mixture of 50% isopropyl xanthate and 50% lignin organosolv reagent is chosen as the collector reagent. The system boundaries for the flotation process include the production of xanthate and lignin organosolv nanoparticles, the transportation of xanthate, the use of xanthate collector in BSC, and the use of lignin organosolv and xanthate collector in S1 for the gold recovery.

Assumptions and limitations

In the LCA method, all materials and energy required during the life cycle of the product or system should be included. However, while conducting the life-cycle inventory and impact assessment it is advised to make assumptions in case of lack of time, resources, and data.

In the current study, regarding transportation, it is assumed that xanthate is purchased and transported from China while the origin country of lignocellulosic biomass is Sweden. The data concerning the components of the flotation system as well as the synthesis processes of the organosolv lignin nanoparticles, such as the quantities of the materials and energy, are confidential; therefore, they are not published in this study.

2.2. Life cycle inventory analysis

In this section, LCI involves the data collection for the production of the OL reagent and the inputs and outputs of the flotation system. The necessary data are acquired from the laboratory process [14].

For the production of organosolv lignin, 8.57 kg of birch wood biomass were added in 85.7 L of solvent containing 50% v/v ethanol and water. The solution was treated at 180 ˚C for 2h by using electricity, without the presence of acid catalyst. At the end of the treatment, two fractions were collected: the cellulose solution and the hemicellulose and lignin solution. Then, lignin was recovered from the pretreated liquor of hemicellulose and lignin by evaporation. After lignin removal, the ethanol solution containing the solubilized hemicellulose is fully recovered and recycled. The fraction containing the cellulose is washed with water.

The inputs and outputs of the system are shown in Table 1 and Figure 1.

Table 1 Inputs and outputs of OL pretreatment

	Flow	Quantity	Unit
Inputs	Utreated birch wood biomass	8.57	kg
	Ethanol	42.85	L
	Water	42.85	L
	Electricity	259.056	MJ
Outputs	Pretreated birch wood biomass	5.23	kg
	Raw organosolv lignin (OL)	1	kg
	Hemicellulose fraction	0.242	kg
	Recovered liquid	85.7	L

The utilisation of organosolv lignin in the flotation process requires the treatment of the lignin in order to obtain the desirable nanoparticles size. The nanoparticles are formed through solvent exchange method. The lignin is dissolved in an ethanol-to-water solution and mixed for 8 hours. Homogenisation is carried out at 750 bars with a pressure homogeniser. For the synthesis of nanoparticles, the solution is diluted with deionised water in a ratio of 1:6 yielding the particles in the form of dry powder. The collection of the particles was accomplished through the lyophilization process.

The inputs and outputs of the gold flotation system for the base case scenario are shown in Table 2 In scenario 1, 50% isopropyl xanthate and 50% organosolv lignin reagent is used as the collector reagent. The inputs and outputs of the gold flotation system for scenario 1 are shown in Table 3.

2.3. Life cycle inventory analysis

In the LCIA phase, the LCI results are associated to environmental impact categories and indicators. The selected impact categories are obtained from ReCiPe 2016, a more extended and updated version of ReCiPe 2008, which was developed by the Royal Netherlands Institute for Public Health and the Environment (RIVM), Radboud University in the Netherlands, the Institute for Environmental Sciences (CML) at Leiden University in the Netherlands, and PreSustainability BV. Endpoint characterisation factors are associated to the areas of protection and obtained from midpoint characterisation factors using a consistent mid-to-endpoint factor for each impact category. The midpoint environmental impact assessment method contains 18 impact categories, from which 11 are examined in this study. The impact categories that will be considered are:

- Climate change,
- Fossil depletion,
- Freshwater ecotoxicity,
- Freshwater eutrophication,
- Human toxicity, cancer,
- Ionizing radiation,
- Land use,
- Marine ecotoxicity,
- Photochemical ozone formation, human health,
- Stratospheric Ozone Depletion,
- Terrestrial Acidification.

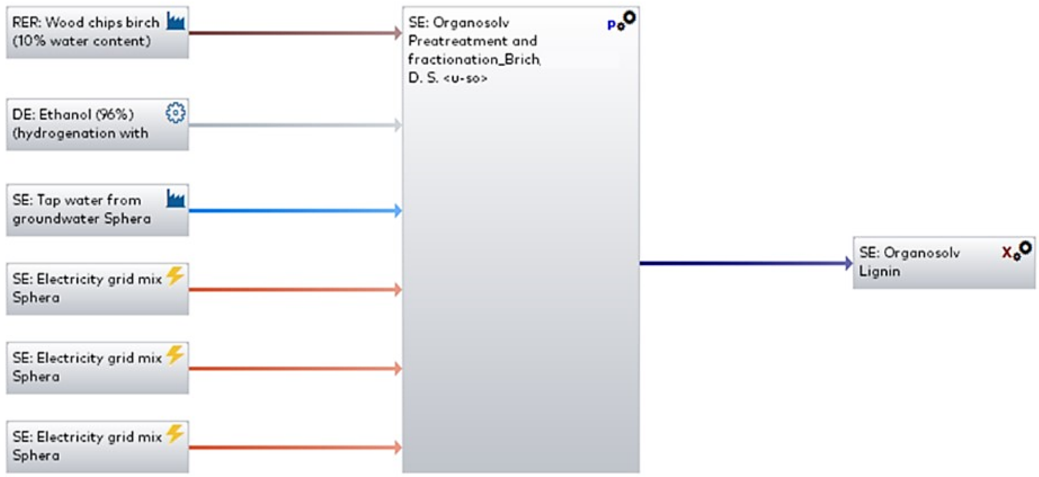


Figure 1 Sphera Model LCA FE software for the pretreatment of birch wood

Table 2 Inputs and outputs of Au flotation system (Base case)

Au flotation system	**Inputs**	Pyrite/ Arsenopyrite + Tailings
		Collector (SIPX)
		Water
		Electricity
		Xanthates transport distance
	Outputs	Pyrite/ Arsenopyrite concentration
		Waste for disposal

Table 3 Inputs and outputs of Au flotation system (Scenario 1)

Au flotation system	**Inputs**	Pyrite/ Arsenopyrite + Tailings
		Collector (SIPX)
		OL nanoparticles reagent
		Water
		Electricity
		Xanthate transport distance
		OL nanoparticles reagents distance
	Outputs	Pyrite/ Arsenopyrite
		Waste for disposal

In order to reflect varying views on time preference, uncertainty or local preference, three different perspectives were created (hierarchist, individualist and egalitarian). The hierarchist perspective is considered the most balanced type as it attains a middle ground between future and present impacts, between risks and benefits, and between local and global concerns. Nevertheless, the hierarchist perspective is applied for the purpose of this study.

3. Results

This section presents and evaluates the results of the life cycle analysis for the investigated system, as well as for all the examined scenarios. Tables 4 and 5 demonstrate the quantified results of BCS and S1 by Sphera Software for each impact category.

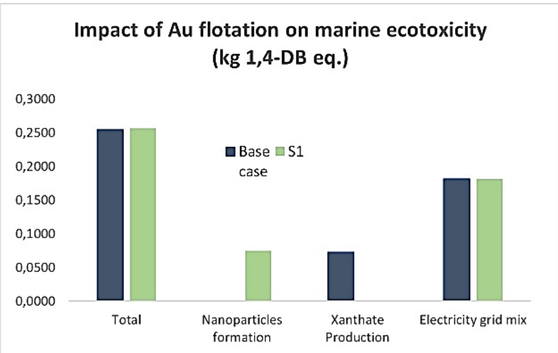
From the outcomes of the two evaluated scenarios presented in the Tables 4 and 5 and the Figures 2, 3, and 4 it is shown that the introduction of organosolv lignin in the production of collecting reagents contributes to the reduction of the environmental footprint of Au flotation. In more detail, from the comparison of the environmental impact values of the base case and scenario 1 circuits, it is estimated that the replacement of xanthate with organosolv lignin nanoparticles reagent shows more environmentally friendly values in all impact categories.

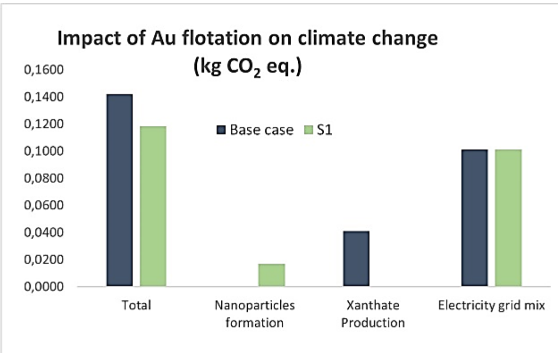
Table 4 Environmental impact values of the Au flotation circuit (base case)

Impact categories	Total	Xanthate Production	Transport (rail)	Pyrite/APy Concentrate	Electricity grid mix	Py/Apy Ore
Climate change [kg CO_2 eq.]	0.1423	0.0409	0	1.41E-05	0.1014	0
Fossil depletion [kg oil eq.]	0.1811	0.0409	0.0713	0	0.0296	0.0305
Freshwater ecotoxicity [kg 1,4 DB eq.]	0.0261	0.0261	0	0	9.60E-06	0
Human toxicity, cancer [kg 1,4-DB eq.]	0.0010	0.0005	0	0	0.0005	0
Ionizing radiation [kBq Co-60 eq.]	0.0910	0.0894	0	0	0.0017	0
Marine ecotoxicity [kg 1,4-DB eq.]	0.2539	0.0725	0	0	0.1814	0

Table 5 Environmental impact values of the Au flotation circuit (Scenario 1)

Impact categories	Total	Nanoparticles formation	Xanthate Production	Transport (rail)	Pyrite/APy Concentrate	Electric grid mix	Py/Apy Ore
Climate change [kg CO_2 eq.]	0.1184	1.03E-04	0.0169	0	1.41E-05	0.1014	0
Fossil depletion [kg oil eq.]	0.1742	9.38E-05	0.0340	0.0713	0	0.0296	0.0305
Freshwater ecotoxicity [kg 1,4-DB eq.]	1.81E-05	3.04E-08	8.50E-06	0	0	9.60E-06	0
Human toxicity, cancer [kg 1,4-DB eq.]	9.15E-04	1.58E-04	0.0003	0	0	0.0005	0
Ionizing radiation [kBq Co-60 eq.]	0.0746	3.32E-05	0.0729	0	0	0.0017	0
Marine ecotoxicity [kg 1,4-DB eq.]	0.2566	0.0749	0.0004	0	0	0.1814	0





Figures 2 and **3** Results of Au flotation measurements on climate change and marine ecotoxicity

A significant improvement is observed in the freshwater ecotoxicity category which is justified by the reduction of the highly toxic xanthate in the aquatic ecosystems. An important difference is also observed in the climate change category with a reduction of 16.79% which is explained by the production of less xanthate compared to the base case scenario. In general, xanthate is associated with greenhouse gas emissions, and therefore the increase in the emitted kg of carbon dioxide (CO_2), justifying the reduction of the value.

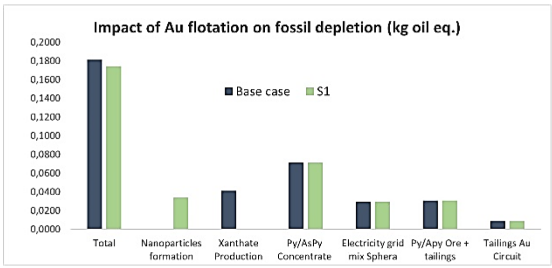


Figure 4 Results of Au flotation measurements on fossil fuel depletion

The value of fossil fuel depletion category is decreased by 3.8% which could contribute to a significant environmental benefit, considering the potential impact on the values on industrial scale processes. The categories of ionizing radiation and destruction of stratospheric ozone show considerably lower values, 18.08% and 24.5% respectively, while the rest of the impact categories show a reduction range from 1% to around 10%. An increase of 1% is detected in the category of marine ecotoxicity which is attributed to the formation of lignin nanoparticles.

4. Discussion

This study aimed to conduct a comprehensive environmental assessment of the use of organosolv lignin reagent in a flotation circuit. The analysis showed that organosolv lignin collectors mixed with xanthate (50% w/w) had a significant improvement in the environmental impact of gold flotation compared to xanthate collectors. In addition to its environmental benefit, lignin can contribute to the support and independence of the European reagents market. With China owning the largest share of production and consumption, the xanthates market size is expected to reach USD 130.5 million by 2027, at a compound annual growth rate (CAGR) of 8.7% during the analysis period, 2020-2027. As a low-cost organic material with almost abundant sources and an increasing global production (expected to reach over $913.1 million by the year 2025) lignin can efficiently replace or limit the xanthate use in the flotation process [15].

Despite its performance in beneficiation methods and its advantages over conventional reagents, the use of lignin-based collectors is only implemented on a laboratory scale. The lack of technical maturity and commercialised solutions poses challenges on the implementation of lignin-based collectors on the mining industry. Regarding the scale-up, many factors affect the process, including the feed, the sizes and design of the industrial cells, the circuit layouts, and the operating conditions of the plant. Experimental data have shown that the approximate flotation rates of ore concentrate recovery in industrial plants are lower than those calculated in laboratory tests. However, the different flotation conditions applied by each plant, as well as parameters such as the type of machines, the operator skills and the feed samples affect the scale-up, making it difficult to adopt a common method of estimating its factors [16].

For the scale-up process of the study, the most efficient method has been proven the estimation of the results by engineering (process simulation, full-scale plant modelling in a virtual environment, calculation of mechanical processes using mass and energy balances) and stoichiometric analysis. A combination of laboratory-scale data and kinetic equations can provide a better approach to industrial results in order to determine the environmental impact [17].

In order to validate these frameworks and to analyse the performance and reliability of the scale-up methods, the laboratory and industrial production processes were investigated. The main idea of all frameworks was to design a scale-up system for the new technology based on similar existing industrial processes. The stages of the scale-up include the simulation of the laboratory and industrial processes, the quantitative description of the new material production and the analysis of the ratios of the scale-up processes [17, 18].

5. Conclusions

This study highlights and evaluates the environmental impacts arising from the flotation process using xanthate reagents, while also examines the environmental benefits of adding organosolv lignin nanoparticles in flotation for the partial replacement of xanthate (50%). The analysis showed that for the production of organosolv lignin, the use of ethanol was the most critical factor contributing to the increase in the environmental impact values, while in the flotation process the main impacts were related to the consumption of electricity and the use of xanthate. In total, the partial replacement of SIPX with OLN resulted in the reduction of 9-10% in the environmental impact of Au flotation. The addition of OLN in the collector mixture and the parallel reduction of SIPX could contribute to the transition to environmentally friendly reagents and therefore to the reduction of the environmental footprint of metallurgical processes.

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NOVA GENERACIJA EKOLOŠKIH REAGENSA NA BAZI NANOČESTICA ORGANOSOLV LIGNINA: PROCENA UTICAJA POSTROJENJA ZA FLOTACIJU ZLATA NA ŽIVOTNU SREDINU

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Izvod

Flotacijska koncentracija je vodeći proces za selektivnu separaciju minerala i obogaćivanje ruda. Princip metode zavisi od različitih osobina kvašenja minerala, tačnije od hidrofobnosti površina koje su ili prirodno hidrofobne ili naknadno postaju dejstvom reagensa. Iako su reagensi široko rasprostranjeni i dugo se koriste, smatraju se veoma opasnim i toksičnim, a njihova razgradnja predstavlja potencijalni rizik po bezbednost, zdravlje i uticaj na životnu sredinu. Nova održiva alternativa koja promoviše održivi razvoj je proizvodnja i upotreba nanočestica organosolv lignina. Ova studija ispituje ekološke prednosti proizvodnje nanočestica lignina od brezovog drveta kao i delimične zamene kolektora ksantata nanočesticama lignina za preradu jedne tone iskopane rude, podvrgnute flotaciji. Kako bi se kvantifikovao uticaj na životnu sredinu, sprovedena je procena životnog ciklusa (LCA) postrojenja za flotaciju zlata. Analiza je pokazala da uvođenje organosolv lignina u flotacijski proces, zajedno sa smanjenjem natrijum izopropil ksantata (SIPX), rezultira smanjenjem uticaja na životnu sredinu, a posebno smanjenjem klimatskih promena i iscrpljivanja fosilnih goriva za 16,79% i 3,8%, respektivno.

Ključne reči: *flotacija, ksantat, lignin, procena životnog ciklusa.*