

An overview of the most actual methods of pyrite ash valorization

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Abstract

The obtaining process of sulfuric acid, that involves roasted of pyrite ores, leads to accumulation of a significant amount of secondary matter known as pyrite ash. This waste, that is found worldwide in quantities of millions of tons, it is highlighted by his chemical composition, based on the presence of high concentrations of hematite and magnetite. By implementing worldwide of the concept of circular economy, the use of pyritic ash as an alternative source of iron is a topic of current interest. This study is proposing to present the latest trends in the methods of valorization of pyritic ash that are presented in the specialty literature, which can be implemented in various branches of industry.

Keywords: *pyrite ash, valorization, metals recovery, value-added products, adsorbent materials*

INTRODUCTION

Pyrite ash is a by-product obtained in the process of burning pyrite ores at temperatures between 600-1000 °C for the production of sulfur dioxide, gas which is processed at the next stage in order to obtain the sulfuric acid used in the chemical reagents industry and chemical fertilizers industry [1].

From a compositional point of view, pyritic ash is a mixture of solid oxides and a small amount of sulfur compounds. The oxide mixture is composed mainly of a significant amount of iron oxides such as magnetite and hematite, but also a small amount of oxides of some metal trace elements (Au, Ag, Cu, Zn, Pb, As) [2]. Due to his chemical content, according to Romanian GD 856/2002, pyritic ash is classified as hazardous waste, with the code 06 01 01 * - wastes from the manufacture of sulfuric acid and sulfurous acid [3]. Table 1 summarizes the chemical composition of pyrite ash, composition that may vary depending on the origin source of the ash.

The production activity of sulfuric acid by the pyritic ores roasting process, took place on a large industrial scale since the 20th century throughout the world, including Romania [4]. Large amount of reddish residue based on pyrite ash, it has been stored over time in open spaces [5]. Thus, over time, could be observed an exponential increase of the amount of pyritic ash stored on land near industrial areas whose object of activity is the production of sulfuric acid. A study conducted by Yanan and collaborators in 2019, disclose that around 15 million tons of pyrite ash are produced worldwide every year, without these being valorized at a later stage. Until the time of the study about 100 million tons of pyrite ash remain unused [6].

Table 1. Chemical composition of pyrite ash

Chemical composition	Concentration	References
Iron oxides (%)	46-46.5	[4], [18], [28]
Cu (%)	0.05-0.79	[4], [17-19]
Co (%)	0.04-0.2	[17], [18], [20]
Zn (%)	0.05-0.58	[4], [18-20], [30]
Au (g/t)	20.55	[19]
Pb (%)	0.02-0.74	[4], [19]
As (%)	0.06-1.52	[19], [30]
S (%)	0.24-1.62	[17-19]
Al ₂ O ₃ (%)	1.91-4.21	[18], [28]
SiO ₂ (%)	1.89-11.05	[18], [28], [30]
CaO (%)	0.59-2.42	[18], [30]

Improper management of significant amounts of pyrite ash has a direct impact on both the quality of the environment and human health. Dispersion of pyrite ash particles in air and soil leads to increased soil acidity, groundwater acidity, rainwater acidity, vegetation loss situated near industrial areas and accelerate the erosion process [7]. Also, the dispersion of pyritic ash in the atmosphere, soil or crops leads to deterioration of human health. Studies present in the literature have shown the high ability of metals to enter the human body in various ways such as: dust inhalation, contaminated with pyritic ash food ingestion and dermal adsorption [8]. The penetration of these metals into the body accelerates some processes for example: lung cancer, skin cancer, kidney damage, cardiovascular disease or severe tiredness [7, 9].

Considering the need to manage large quantities of pyrite ash deposited on the ground in open space, the considerable quantitative decline regarding metallic natural resources over the last decades [10], but also their influence of the environmental quality and human health, the researchers have tried over time to develop different methods for pyritic ash valorization.

MATERIALS AND METHODS

The depletion of natural resources over time is one of the major humanity problems today. To reduce this problem the concept of circular economy has been introduced which involves the repair, reuse, and recycling of materials, products and waste as long as possible [11].

Scientists have tried pyrite ash valorization in various areas of industry. The main valorization method of this waste, was implemented at industrial scale, was the use as a raw material in the steel industry by pelletizing [12]. Due to the large amounts of pyritic ash existing worldwide, researchers have tried to develop alternative valorization pyrite ash methods, these being used in building materials industry, ceramics industry, pigments industry, catalyst industry and adsorbent materials industry and in chemical looping combustion processes. Figure 1 shows the main current methods of pyrite ash valorization.

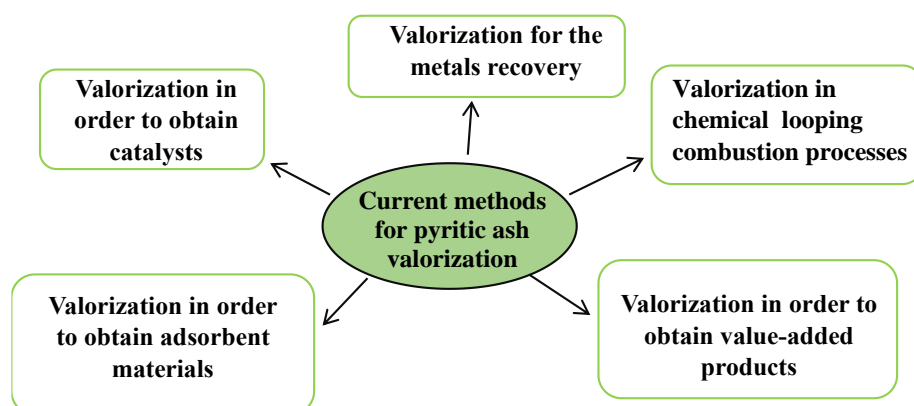


Fig. 1. Current methods for pyritic ash valorization

RESULTS AND DISCUSSIONS

Valorization of pyrite ash in order to recover metals

One of the most used method for pyritic ash valorization in order to recover the metals from their composition is the acid leaching method and the bioleaching method with the help of microorganisms.

Acid leaching method is one of the most widely used methods for pyritic ash valorization. This method is based on the metals leaching, that are in the composition of various ores, with the help of acid solutions. The most commonly used reagents in the acid leaching processes are sulfuric acid and phosphoric acid. The advantages of acid leaching method include the rapidity, ease of implementation and the high ability to control the process [13].

Due to the high iron content, which is present in the composition of pyritic ash, the latest studies presented in the literature focus on pyritic ash valorization in order to iron and minor components such as Cu, Co, As, Zn simultaneously recover. The efficiency of the iron from pyritic ash separating process is difficult to achieve due to fine granulation of pyrite ash, but also to the strong interaction between iron atoms and toxic heavy metal atoms [14]. Over time, studies that involving iron separation from pyrite ash have focused on physical methods based on magnetic separation [7] and gravitational separation [15]. The physical separation processes yields of iron from pyrite ash were low (<65%). In order to improve the leaching efficiency of iron from pyritic ash, researchers combined physical separation methods with chemical methods based on the interfering ions extraction and precipitation [16].

The researchers team formed by Zhang and collaborators [17] published in 2021 in the literature a study about leaching and iron separation from pyritic ash. Because previous research has shown that the copper ions presence in pyritic ash composition has a negative influence of in the iron separation process, during the study conducted by Zhang and his team attempted removal this disadvantage. The experimental steps consisted of a preliminary step of reducing the pyrite ash samples to 800 °C, a copper acid leaching step, followed by a magnetic separation step of the iron from the residue remaining from the leaching process. Experimental results obtained by the research team by optimizing important parameters such as sulfuric acid concentration, pyrite ash particle size, temperature and stirring speed, demonstrated a trend of increasing iron recovery rate from pyrite ash up to 96.99% when an additional copper extraction step is implemented from the sample, with copper extraction yield exceeding 80%.

Studies also suggest the possibility of simultaneous recovery of three metals present in the composition of pyritic ashes. Thus, in the study published in the literature by Jiang Tu and co-workers [18], the pyritic ashes have been exploited for the simultaneous leaching of iron, copper and cobalt. The process of recovering the three metals comprises three stages: preliminary sample preparation step, leaching step in phosphoric acid to extract cobalt and copper ions and precipitate iron ions as iron phosphates and an extraction step of cobalt and copper ions with P₂O₄. The experimental results obtained demonstrated a direct influence of temperature, acid concentration, solid: liquid ratio on the acid leaching process of the three metals from pyritic ash. The recovery yields of Cu, Co, Fe increased to 82%, 99%, 98% respectively.

The acid leaching recovery method is also used for the recovery of gold in pyritic ash waste. In order to eliminate the negative influence of the presence of arsenic on the efficiency of the gold extraction process, ores containing both gold and arsenic undergo a chemical pre-treatment step to remove arsenic ions before the actual gold extraction step takes place. The study by Wang and co-workers [19] aimed to valorization pyritic ash for gold leaching. The experimental results reported an increase gold extraction yield when used cyanide-free agent to about 66% and the experiment is accompanied by the acid pretreatment step of the pyritic ash sample. Compared to the situation when gold is extracted directly from the pyritic ash without the acid pretreatment step, the final amount of gold extracted, in this case, from the residue being 7.4 g/t.

Due to the main disadvantages of acid leaching, including high pollution, high costs and increasingly stringent environmental laws, it has become essential to use environmentally friendly methods to recover minerals [20]. In recent decades, the extraction of certain chemical

compounds/elements from various industrial wastes using microorganisms by bioleaching method experienced has become increasingly used a significant growth. The main advantages of this methods are: reduced degree of pollution and contamination, significantly lower costs, ability of microorganisms to tolerate higher concentrations of heavy metals, but also the ability of microorganisms to grow and develop in extreme acidic pH conditions [21]. A study by a team of researchers [20] reports the use of bioleaching for the simultaneous recovery of four metals (Co, Cu, Zn, As) that are present in the composition of pyritic ashes. The study used a mixture based on three classes of autotrophic aerobic microorganisms. The aqueous extraction yield for arsenic and zinc increased by more than 60%, while the aqueous extraction yield for copper and cobalt increased by more than 90% [20].

Valorization of pyritic ashes in the chemical looping combustion process as oxygen carrier

The greenhouse effect from the burning of fossil fuels is one of the sources of global warming and represent one of today's most serious environmental problems [22, 23]. To mitigate greenhouse effects, efforts have been made to develop and improve technologies to reduce CO₂ emissions worldwide. Chemical looping combustion technology has gradually become an alternative technology for capturing and storing carbon dioxide from fossil fuel combustion [24]. To date, about 1200 types of optimal oxygen carriers are known to be used in the chemical loop combustion process, such as iron, copper, nickel or cobalt oxides. In order to reduce the cost of synthesizing oxygen carriers, attempts have been made to use a variety of wastes as oxygen carriers, including pyritic ash [25-27]. In order to be able to apply in chemical loop combustion technology on an industrial level, optimal oxygen carriers must be developed that present the following characteristics: high redox performance, high reaction rates, high stability, mechanical strength, low costs and ease of manufacture.

Due to the chemical composition of pyritic ash-based wastes, especially the presence of Fe₂O₃, Zhong and co-workers attempted to exploit pyritic ashes as oxygen-carrier materials for the chemical looping combustion process [28]. In this study, was investigated the reactivity and cycling stability of two pyrite cinder samples (a sample of pyrite cinder with rich Fe₂O₃ and a sample of pyrite cinder with rich inert constituents and CaSO₄) in a fixed-bed reactor under different reaction temperatures. Comparative studies have shown that the oxygen carrying capacity for pyrite cinder with rich inert constituents and CaSO₄ sample maintained at around 2.73% after several redox cycles and at the same conditions the oxygen carrying capacity of pyrite cinder with rich Fe₂O₃ sample was about 2.45%. Furthermore, it was observed that pyrite cinder with rich Fe₂O₃ sample suffered serious deactivation and surface sintering in the multiple cycle experiment while the pyrite cinder with rich inert constituents and CaSO₄ sample presented excellent redox performance and anti-sintering ability. Experimental results indicated that pyrite cinder rich in CaSO₄ and inert components showed promising redox performance in redox cycles at chemical looping combustion processes.

More, comparative studies relating to activity and redox stability between pyrite cinder sample and two natural Fe-based oxygen carriers were carried out by Zhong Ma et al, in 2020 [25]. In the study, pyrite cinder was used as an oxygen carrier in CH₄ chemical looping combustion in a fixed-bed reactor using CH₄- N₂ reducing gas. The experimental results obtained demonstrated that pyritic ash samples show a high anti-sintering ability during 20 redox cycles. The analysis of studies related to the redox performance of the oxygen carriers for the pyrite cinder, hematite and ilmenite was demonstrated that the CH₄ conversion and CO₂ selectivity for hematite decline after the first redox cycle, in contrast with pyrite cinder and ilmenite, which both could maintain the redox reactivity in the multiple cycles. Also, comparative studies between pyrite cinder, synthetic Fe-Al(10nm) oxygen carrier and natural MAC iron ore capacity as oxygen carriers in chemical looping combustion were carried out by Zhang S. and co-workers [26]. The cyclic reactivity and attrition tests showed that pyrite cinder and MAC iron ore possessed a strong resistance to the reactivity and physical structure decay with the redox cycles proceeding, in opposition with synthetic Fe-Al(10nm) oxygen carrier.

Valorization of pyritic ashes into value-added products

Given the concern for environmental protection in recent years, pyritic ash-based wastes have started to be reused for cost-effective with high value-added products used in the ceramics industry [29] or in the building materials industry [30,31].

By comparative compositional analysis of ceramic products and pyritic ashes, it was found that both products have a high content of iron, silicon and aluminum oxides. Based on this observation, the study developed by Zhang and co-workers assumed the use of pyritic ashes as a source of iron in the composition of ceramic products, thus obtaining value-added products [29]. The study consisted of an intermediate stage of processing the pyritic ashes, followed by a stage of homogenization with coal powder and calcium fluoride and finally casting into shapes. Subsequent tests carried out on the synthesized materials have demonstrated the significant influence of physicochemical parameters such as: temperature and time of synthesis, dose and type of additive, casting pressure, on the quality of the value-added products obtained, an aspect supported by other results in the literature [32, 33].

Also, due to the high content of iron oxides, found in the form of magnetite and hematite, pyritic ash has been exploited in the building materials industry as an alternative source of iron [30, 31]. A study about utilization of pyritic ashes in cement production at industrial-scale has been reported in the literature by Alp et al [30]. They followed the evolution of mechanical, physical and chemical properties of building materials sintered from pyritic ashes for a period of 6 months. The results obtained were compared with those of similar materials made from natural iron ore. The research team observed a significant improvement in the setting time of mortar obtained from pyritic ash compared to the setting time of mortar based on natural iron ore, while the compressive strength did not differ significantly.

In addition, the presence of hematite in the composition of pyritic ashes has allowed their use for the synthesis of iron oxide pigments [34-36]. In the literature study by Zheng and co-workers, pyritic ash waste was used as a secondary raw material for the preparation of micaceous iron oxide pigment [36]. The study included leaching, precipitation and hydrothermal treatment stages. The results obtained by the research team demonstrated the direct influence of some physical-chemical parameters such as NaOH concentration, total iron concentration, reaction temperature and reaction time on the morphology and color of the synthesized pigments. The quality tests carried out by the research team indicated that the synthesized pigments required characteristics of micaceous iron oxide pigments for paints.

Valorization of pyritic ash in order to obtain adsorbent materials and zeolites

Another side of interest regarding the valorization of pyritic ashes is their use in the composition of adsorbent materials. Materials with adsorptive properties have arisen through the combination of high magnetic field separation capacity and enhanced adsorption capacity through functionalization [37]. These types of adsorbent materials have the advantage of easy separation, due to their magnetic properties, when used as adsorbent materials for adsorption of different ions from aqueous solutions. Based on the high iron content of pyritic ashes, which gives them magnetic properties, the research team of Li and collaborators [38] synthesized adsorbent materials based on manganese dioxide deposited on the surface of pyritic ashes, which were then used in the process of retaining thallium ions from wastewater. The experimental results demonstrated the maintenance of long-term adsorption properties, the adsorption properties of the material based on pyritic ash and MnO₂ remained unchanged after five consecutive regeneration cycles with 1M HCl, directly proportional increase between the adsorbent material surface area and efficiency of the thallium ions adsorption process. More, adsorption properties of the new adsorbent material remained high throughout the pH range 2-12. Another study conducted by Turk, in 2016, studied pyritic ash valorization as a potential adsorbent for removing arsenic (As) from groundwater. The results indicate that adsorption process of As by pyritic ash was affected by initial pH, amount of adsorbent added over the range of 0.1–50 g/L and the As(V) removal increased with time (79 % removal was achieved within 1 h). Experimental results obtained have demonstrated the high capacity of pyritic ash to

adsorb arsenic from wastewater, approximate 97 % of the As was adsorbed at an adsorbent dosage of 10 g/L, a solution pH of 9, a temperature of 25 C, and an initial As concentration of 300 µg/L [39].

In addition, this type of waste was used to prevention of air pollution with sulfur compounds. In the study presented by Dancila and co-workers pyritic ash waste was used in the composition of sorbets materials for retention H₂S, from fuel gases. The study presented by Dancila and co-workers consisted in the preparation of three sorbents based on pyritic ashes and a zinc wurtzite oxide, in different rations to obtain a mixed ZnFe₂O₄ oxide. To test retention capacity of the three sorbents were saturated with H₂S and it was noticed that the retention capacity increased once the molar ratio of Fe₂O₃ from the pyritic ash samples increased [40].

Valorization of pyritic ashes due to their magnetic properties has also targeted the field of zeolites. Due to the main disadvantage represented by the reduced separation capacity of zeolites due to the small size of the zeolite particles, the development of some zeolitic materials with magnetic properties has been attempted. The magnetic properties confer high extraction capacity of zeolites from solution by the magnetic separation process. By combining the adsorbent properties of zeolites with the magnetic properties of pyritic ash, the research team coordinated by Wang synthesized, using the hydrothermal method, a new zeolitic adsorbent material based on kaolinite-enriched pyritic ash [41]. Due to its chemical composition, kaolinite was silicon and aluminum source necessary for the zeolite preparation. The developed material was used in the treatment of wastewater by adsorbing the Cs⁺ ions present in their composition. The experimental results obtained revealed a good adsorption capacity of the new material developed for Cs⁺ ions.

Valorization of pyritic ash in order to obtain catalysts

The presence of iron oxides in the composition of pyritic ashes makes these residues remaining from the pyrite ore combustion process a promising alternative for use as catalysts in the catalytic ozonation process. In order to increase the efficiency of the catalytic ozonation process for the degradation of toxic organic compounds, the use of catalysts based on transition metal ions (Co²⁺, Ti²⁺, Mn²⁺, etc.) has been reported in the literature [42,43]. Subsequently, catalytic ozonation studies on the degradation of wastewater phenols in which the catalyst was Fe₂O₃ were reported in the literature [44]. Wu et al. conducted a study in which pyritic ashes were used in the heterogeneous catalytic ozonation process to degrade the reactive black 5 (RB5) in wastewater [45]. Black 5 reagent belongs to the class of synthetic dyes used in the textile industry in fabric dyeing processes. Studies show that about 12-14% of the composition of dyes used in dyeing processes is released into the effluent [46]. The study carried out by Wu et al. tested the catalytic properties of unmodified pyrite ash and catalysts based on pyrite ash and synthesized Ce³⁺ ions. The unmodified pyrite ash catalyst accelerates the RB5 degradation process by about 18%, while the pyrite ash and Ce³⁺ catalyst significantly improves the degradation process by about 42%. At the same time, studies have shown that the two catalysts tested exhibit stable catalytic activity in the pH 3-10 range [45].

CONCLUSIONS

This review of the literature about pyritic ash valorization showed that the most current studies have been focused on the application of the circular economy principles. Many of studies present in the specialized literature have demonstrated the use of pyritic ash as source of metals extraction, such as Fe, Cu, Co, Zn, As, Au, thus protecting exhaustible natural ore resources. On the other hand, due to the presence of iron in chemical composition of this waste, pyrite ash has been used in many studies as raw material in processes from different industrial branches. The presented studies demonstrate the high capacity of pyritic ash to be used in composition of ceramic materials, building materials and pigments. Also, pyrite ash helped solve some environmental problems, such as air and water depollution, being used as catalyst, adsorbent material or oxygen carrier in the chemical looping combustion. Finally, it is remarkable the exploitation and application potential of high quantities of pyritic ash at industrial scale in numerous fields.

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