THE COMBINED PROCESSES FOR REMOVING INORGANIC SULPHUR FROM COAL IN ORDER TO DIMINISH THE SO_2 EMISSION IMPACT ON THE ATMOSPHERE

CISMAŞIU Carmen-Mădălina, TOMUŞ Nicolae, CIOBOIU Olivia, POPEA Florina, TONIUC Maria, DEÁK Ştefania-Elena

Abstract. The increased in environmental pollution determined an enhanced interest in the microbial resistance to heavy metal ions and especially on the extension of the potential of biotechnological applications of acidophilic microorganisms. Physical methods for coal desulphurization developed nationally and internationally, involve the separation of pyrite (FeS_2) from coal, by flotation processes, hydrogravitational processes, and magnetic separation. The case study was conducted for crude coal taken from mining operations in the Valea Jiului coal basin, Romania. In order to determine the content of granulometric classes of less than 1 mm, analyses were performed for coal from the Vulcan, Lupeni, Lonea, Paroşeni and Petrila mines. The distribution of sulphur on densimetric fractions for coal in the Jiu Valley is on average 1.9% and different in coal fractions, for example: 2.65% for densimetric fractions -1.36 t / m^3, 1.36 ÷ 1.73 t / m^3; 1.19% for the densimetric fractions 1.73 ÷ 2.2 t / m^3 and +2.2 t / m^3. The physical processes for removing inorganic sulphur from coal are: (1) concentration in hydrogravitational field - sulphur extraction was 25 ÷ 30% in the concentrate and mixtures resulting from the separation, with the Gemini mass. Also, there was an improvement in the quality of coal, by increasing the ppc content in the table waste, but also a loss of coal in the mixture from gravitational separation, up to 15% ÷ 20%; (2) the separation in magnetic field - content of sulphur in the magnetic product, extracted with the lift-magnet separator, for mining exploitation perimeters was: 11.48% Lupeni; 8.34% Paroşeni; 6.4% Vulcan; 5.85% Lonea and 3.09% Petrila; and the sulphur content in the non-magnetic product (76-94%) was: 1.14% Petrila, 1.12% Lonea, 0.57% Vulcan, 0.51% Paroşeni and 0.48% Lupeni. In the non-magnetic product (desulphurized coal) there is still between 40-54% of the sulphur existing in the original coal, which means that sulphur is not only related to pyrite, but also to sulphates without magnetic properties.

Keywords: coal, inorganic sulphur, bacterial oxidation, ecological valence, south-western Romania.

Rezumat. Procedee combinate de îndepărtare a sulfului anorganic din cărbune în scopul diminuării impactului emisiilor de SO_2 în amosferă. Accentuarea gradului de poluare a mediului a determinat creşterea interesului cu privire la rezistenţa microbiană la ioni de metale grele şi mai ales asupra extinderii poteanţialului de aplicaţii biotehnologice a microorganismelor acidofile. Metodele fizice pentru desulfurarea cărbunilor, dezvoltate la nivel naţional şi internaţional, presupun separarea piritei (FeS_2) de cărbune, prin procese de flotaţie, procedee hidrogravitaţionale şi separare magnetică. Studiul de caz s-a realizat pentru cărbunele brut prelevat de la exploatările miniere din bazinul carbonifer Valea Jiului, România. În scopul determinării conţinutului de clasă granulometrică mai mică decât 1 mm, s-au realizat analize pentru huila de la minele Vulcan, Lupeni, Lonea, Paroşeni şi Petrila. Repartiția sulfului pe fracțiuni densimetrice la cărbunele din Valea Jiului este în medie de 1,9% și diferită în fracțiunile de cărbune, de exemplu: 2,65% pentru fracțiunile densimetrice -1,36 t/m^3, 1,36 ÷ 1,73 t/m^3; 1,19% pentru fracțiunile densimetrice 1,73 ÷ 2,2 t/m^3 și +2,2 t/m^3. Procedeele fizice de îndepărtare a sulfului anorganic din cărbine sunt: (1) concentrarea în câmp hidrogravitațional - extracția de sulf a fost de 25 ÷ 30% în concentratul și mixtele rezultate în urma separării, cu masa Gemeni. De asemenea, s-a remarcat şi o îmbunătăţire a calităţii cărbunelui, prin creşterea conţinutului de ppc în sterilul mesei, dar şi o pierdere de cărbune în mixtul de la separarea gravitaţională, de până la 15% ÷ 20%; (2) separarea în câmp magnetic - conţinutul de sulf în produsul magnetic, extras cu separatorul liftmagnet, pentru perimetre miniere de exploatare a fost: 11,48% Lupeni; 8,34% Paroşeni; 6,4% Vulcan; 5,85% Lonea şi 3,09% Petrila; iar conţinutul de sulf în produsul nemagnetic (76-94%) a fost de: 1,14% Petrila, 1,12% Lonea, 0,57% Vulcan, 0,51% Paroşeni şi 0,48% Lupeni. În produsul nemagnetic (cărbunele desulfurat) a mai rămas între 40-54% din sulful existent în cărbunele iniţial, ceea ce înseamnă că sulful nu este legat numai de pirită, ci și de sulfaţi fără proprietăţi magnetice.

Cuvinte cheie: cărbune, sulf anorganic, oxidare bacteriană, valențe ecologice, sud-vestul României.

INTRODUCTION

The study is part of a project based on physical methods for the desulphurization of coals, developed at national and international level, which supports responsible actions to reduce greenhouse gas emissions during the transition to an economy without fossil fuels. The unique properties of extreme microorganisms make them useful for a number of biotechnological applications in a variety of industrial and environmental protection. The ecological approach to biotechnology based on the biodiversity of microorganisms involves investigating as many natural environments as possible and, in particular, different habitats of the type of extreme environments (CIOBOIU & CISMAȘIU, 2016; CIOBOIU et al., 2017; CISMAȘIU & CIOBOIU, 2021; MURARIU & MAICAN, 2021).

The development of biotechnological processes, based on the activity of acidic microorganisms, demonstrated their efficacy in removing metallic ions in mining effluents with two beneficial effects: (1) bioremediating polluted environments with residual inorganic substances, such as heavy metals that are toxic for most forms of life, including man; (2) recovery of some quantities of metallic ions for their reuse for economic purposes (DEAK & DEAK, 2005; TOMUȘ & CISMAȘIU, 2014; GAVRILESCU et al., 2018; HAFEEZ et al., 2019; MURARIU & MAICAN, 2021).

Globally, it has been shown that the biodegradative action of acidic bacteria has a major contribution to the circulation of biological elements in nature, and locally prevents the accumulation of residual materials of the various contaminants of the environment. The development of biotechnological processes, based mainly on the activity of acidophilic microorganisms, has demonstrated their effectiveness in the recovery of metals from sulphur minerals and mining drainage, as well as to the bioremediation of the polluted environment with residual inorganic substances, such as heavy metal ions and their compounds (HAKI & RAKSHIT, 2003; JOHNSON & HALLBERG, 2008; VOICU et al., 2009).

The increase of environmental pollution has sparkled interest in microbial resistance to metallic ions and especially in the expansion of bio-technological applications of acidophilic microorganisms. Physical methods for the desulphurization of coal, developed at national and international level, involve the separation of pyrite (FeS_2) of coal, by floating processes, hydrographic processes and magnetic separation (JOHNSON, 1999; VOICU et al., 1999; KATYAL & PETRIŞOR, 2012; ZHU et al., 2022).

MATERIAL AND METHOD

Two types of coal were used in the bacterial desulphurization experiences of coal by bacterial cultures of *Acidithiobacillus (Thiobacillus) ferrooxidans*: (1) pit coal from Lupeni and (2) lignite from Turceni. In order to study the increase in the yield of the bacterial oxidation process of the inorganic sulphur in coal, the experiments were accompanied by chemical and biological witnesses. At the end of the experimental period, the weight loss of coal was determined under the action of *Acidithiobacillus (Thiobacillus) ferrooxidans*, which rendered soluble the pyrite by transforming the ferrous iron from the sulphide into soluble ferric sulphate. Also, in the treated coal samples, the amount of inorganic sulphur was determined by the gravimetric method with $BaSO_4$ at the Chemistry Laboratory of the Research and Development National Institute for Radioactive Metals and Resources. Laboratory experiments and analyses were carried out in: (1) the laboratory of mineral resources techniques and technologies, Department of Research at the Research and Development National Institute for Metals and Radioactive Resources - ICPMRR Bucharest, (2) the laboratory of chemical and structural analyses, Research Department of the Research and Development National Institute for Metals and Radioactive Resources - ICPMRR Bucharest, (3) the Department of Microbiology, Bucharest Institute of Biology of the Romanian Academy.

RESULTS AND DISCUSS

Research shows that the best results in coal desulphurisation by combined methods have been obtained by removing up to 38% of sulphur by intensive magnetic field separation in dry and wet environments (accompanied by the removal of sulphur part of the coal-bearing iron ore), combined with the action of acidophilic chemoautotrophic bacteria of the type *Acidithiobacillus (Thiobacillus) ferrooxidans*, which favoured a further reduction of the inorganic sulphur content by up to 70% of the content of coal when entering this process of controlled bacterial bio-oxidation. Experiments of oxidation of coal concentrates with cultures of *Acidithiobacillus* (*Thiobacilus*) *ferrooxidans* at different solid / liquid ratios showed that, out of the 5 solid/liquid ratios tested, at a solid density of 4g/100ml and 5g/100ml, an increased yield of biooxidation of inorganic sulphur was obtained from coal, reaching a percentage of 65.31% - 74.23% for pit coal and 78.88% - 88.46% for lignite, at the same time of experimentation (CHEMYSH et al., 2019; GARG et al., 2020; GAVRILESCU et al., 2020; HAFEEZ et al., 2020; MISHRA et al., 2020; MELENDEZ-SANCHEZ et al., 2021; MURARIU & MAICAN, 2021).

A) Removal of inorganic sulphur from coal by physical processes. The research has shown that significant results were obtained by two types of processes: (1) the hydrographic field concentration: sulphur extraction was 25 ÷ 30% in the concentrate and mixes resulting from the separation with the Gemini table (Fig. 1). An improvement in the quality of coal was also noted, by increasing the PPC content in the tab letter, but also a loss of coal in the mixture of gravity separation, up to 15% ÷ 20%, (2) magnetic field separation: the content of Sulphur in the magnetic product, extracted with the lift-magnet separator (Fig. 2), for mining perimeters of exploitation was: 11.48% Lupeni; 8.34% Paroşeni; 6.4% Vulcan; 5.85% Lonea and 3.09% Petrila; and the sulphur content in the non-magnetic product (76-94%) was: 1.14% Petrila, 1.12% Lonea, 0.57% Vulcan, 0.51% parents and 0.48% Lupeni. In the non-magnetic product (desulphurated coal) there was between 40-54% of the existing sulphur in the initial coal, which means that the sulphur is not only bound by pyrite but also sulphates without magnetic properties.

B) The study of possibilities of inorganic desulphurization of coals by combined processes. Physical desulphurization analyses have shown that none of the test procedures significantly diminishes the sulphur content in the analysed coal so as to justify the related expenses. The results of experiments recommend coal desulphurisation through combined methods between physical processes, respectively the separation of coal pyrite and microbiological leaching process, using acidophilic acid, iron- and sulphur oxidizing bacteria, *Acidithiobacillus (Thiobacillus) ferrooxidans*. The technology comprises the grinding operation, followed by physical concentration processes represented by the magnetic separation combined with the hydrographs process and then the bacterial solubilization process in order to remove inorganic sulphur in the coal samples taken in the study (Fig. 3).

Figure 1. Gemini concentration table (original).

Figure 2. Lift-magnet magnetic separator type Leneigrad (original).

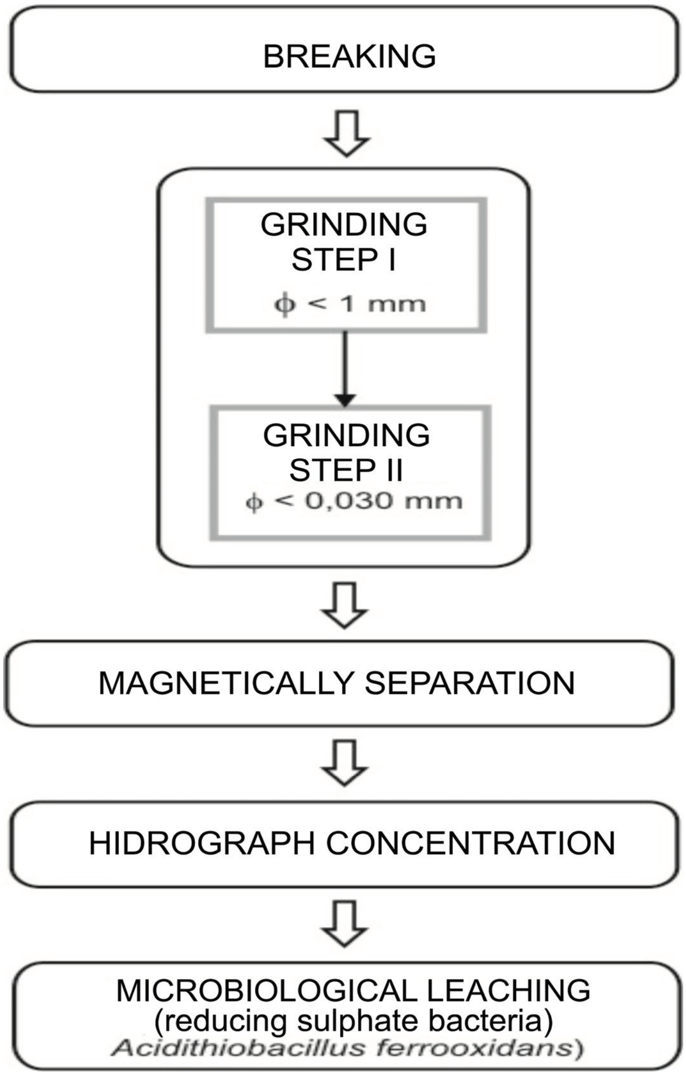

Figure 3. Technology operations of coal desulphurization
by combined physical-microbiological methods in the premptors phase (original).

In the experiences of desulphurizing coal, bacterial cultures of *Acidithiobacillus (Thiobacillus) ferrooxidans* were used (9 populations), isolated from the Sesei Valley (Alba County) and Baia (Tulcea County), selected based on increased concentrations of iron (18g/l), copper and zinc (3000ppm). In the experiences of bacterial desulphurization of coal by bacterial cultures of *Acidithiobacillus* (*Thiobacillus*) *ferrooxidans*, two types of coal were used: (1) pit coal from Lupeni and (2) lignite from Turceni. Coal concentrates taken in the study were used as energy substrates for increasing the cultures of *Acidithiobacillus (Thiobacillus) ferrooxidans* in Leathen medium. In the sulphur biooxidation experiences, a solid / liquid ratio of 1-5g/100ml in Leathen medium, coal granulometry was 0.75mm. The bacterial inoculum was the cultures of the population of *Acidithiobacillus ferrooxidans* raised in Leathen's medium, where the energy substrate was coal. The end of the experimental period was determined by weight loss of coal under the action of *Acidithiobacillus (Thiobacillus) ferrooxidans*, which solubilizes pyrite by transforming ferrous iron from sulphide into soluble ferric sulphate. Also, the treated coal samples were used to determine the amount of inorganic sulphur by the gravimetric method with $BaSO_4$ at the Chemistry Laboratory at the National Research and Development Institute for Metals and Radioactive Resources.

Cultures of *Acidithiobacillus* (*Thiobacillus) ferrooxidans* used in the sulphur biooxidation experiences of the tested coal concentrates were selected based on ferrous sulphate oxidation capacity in the presence of high metal ions. Thus, in order to increase the efficiency of bacterial desulphurization processes of coal concentrates, P_3 population of *Acidithiobacillus (Thiobacillus) ferrooxidans* were selected as a reference population due to its increased sensitivity at high metal ions in culture medium. Sulphur biooxidation experiences from Lupeni coal concentrates in various solid / liquid ratios (1-5g/100ml) were carried out in the presence of *Acidithiobacillus* (*Thiobacllus) ferrooxidans* in continuous stirring conditions and are shown in figures 4 and 5.

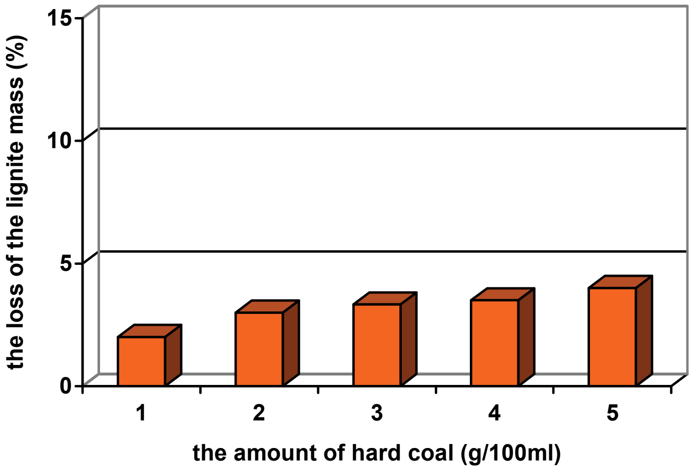

Figure 4. Variation in weight loss of coal samples in the presence of the P_9 population of *Acidithiobacillus (Thiobacillus) ferrooxidans*, tolerant to 8g/l ferrous sulphate.

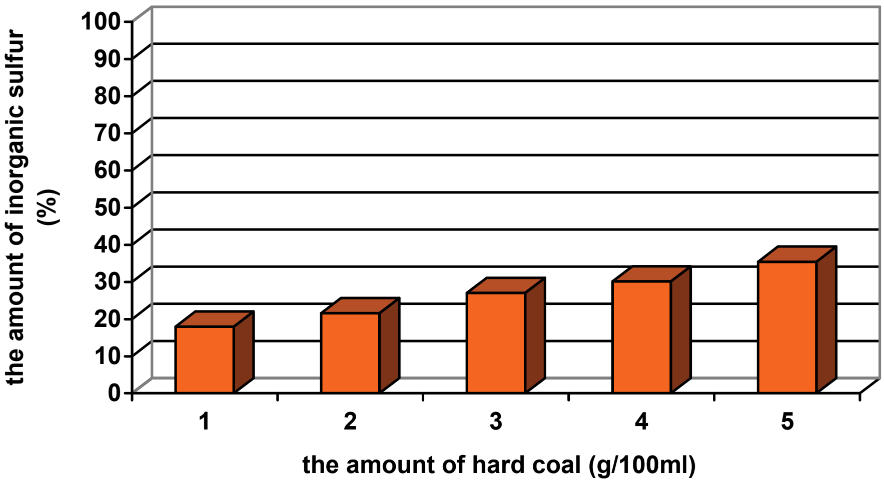

Figure 5. Variation of inorganic sulphur content in pit coal samples in the presence of the P_9 population of *Acidithiobacillus (Thiobacillus) ferrooxidans*, tolerant to 8g/l ferrous sulphate.

After testing the sulphur biooxidation capacity of the coal concentrate at a solid density of 4g/100ml and 5g/100ml, it was found that the P_5 and P_7 populations determined an increased efficiency of the coal desulphurisation efficiency in the same time interval, a consequence of the tolerance raised to ferrous sulphate in solution of these cultures. It was also found that at the density of the solid of 5g/100ml an increased efficiency was obtained in the oxidation of inorganic sulphur in coal, evidenced by obtaining smaller amounts of inorganic sulphur in it, which reach values between 48,12-74,23% (Figs. 6; 7).

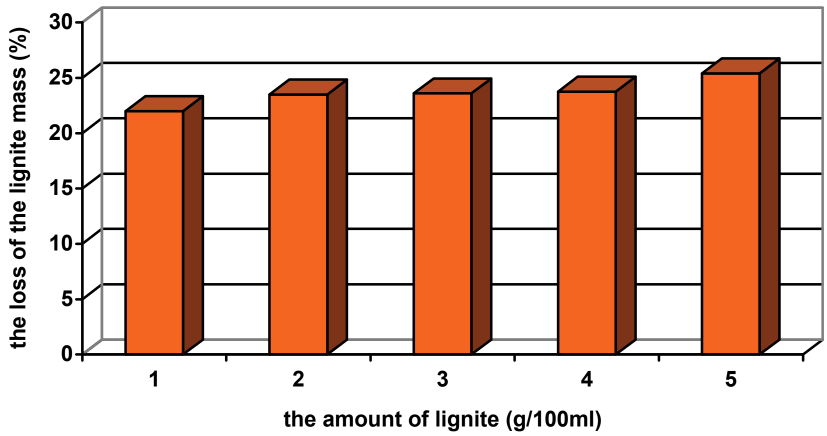

Figure 6. Variation in weight loss of lignite samples in the presence of the P_7 population of *Acidithiobacillus (Thiobacillus) ferrooxidans*, tolerant to 18g/l ferrous sulphate.

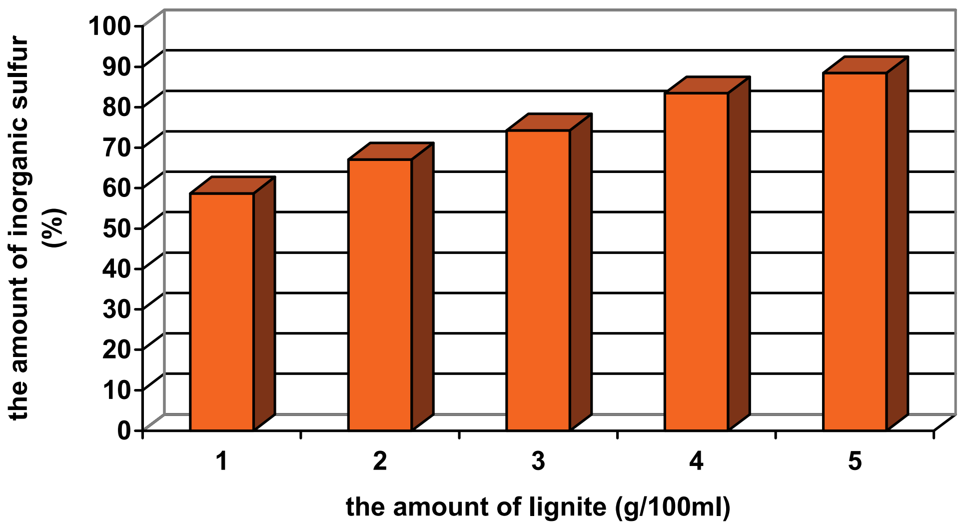

Figure 7. Variation of inorganic sulphur content in lignite samples in the presence of the P_7 population of *Acidithiobacillus (Thiobacillus) ferrooxidans*, tolerant to 18g/l ferrous sulphate.

CONCLUSIONS

The accumulation of stimulant products and antagonistic phenomena contributes to the qualitative and quantitative change of nutrients in the culture medium, as well as the occurrence of changes in the structure of communities of microorganisms that underlie the succession of populations in an abiotic system. Extracellular enzyme production by some members of natural microorganism communities, often as dense populations, is relatively stable and difficult to disrupt. These data confirm the increased resistance of acidophilic bacterial species to extreme environmental conditions and the accumulation of metal ions in the environment depending on their concentration. In this regard, the data obtained illustrate that acidophilic microorganisms introduce non-native substances into the food web, modify the values of pH, t^0, Eh through oxidation-reduction reactions, degrade "recalcitrant" substances. Acidophilic microorganisms also assimilate a wide range of chemicals and reintroduce substances that are inaccessible to other microorganisms into the food web by acting as geochemical agents.

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Cismașiu Carmen-Mădălina, Popea Florina, Toniuc Maria
Institute of BiologyBucharest, Romanian Academy, Spl. Independentei No. 296, sect. 6, 060031, Bucharest, Romania.
E-mails: carmen.cismasiu@ibiol.ro, carmencismasiu@gmail.com.

Tomuş Nicolae
Research and Development National Institute for Metals and Radioactive Resources (INCDMRR), Ilfov, Romania.
E-mail: icpmrr@icpmrr.ro

Cioboiu Olivia
The Oltenia Museum, Craiova, Str. Popa Șapcă, No. 8, 200422, Craiova, Romania.
E-mails: oliviacioboiu@gmail.com; cioboiu.olivia@yahoo.com

Deák Ştefania-Elena
CIT-IRECSON Bucharest, Romania.
E-mail: stef.deak@yahoo.com

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