UNDERGROUND MINING ENGINEERING

Podzemni radovi



University of Belgrade – Faculty of Mining and Geology

	ISSN	0354-2904
UDK 62	eISSN	2560-3337

UNDERGROUND MINING ENGINEERING

PODZEMNI RADOVI

N°42.



http://ume.rgf.bg.ac.rs e-mail: editor.ume@rgf.bg.ac.rs

Belgrade, June 2023.

UNDERGROUND MINING ENGINEERING 42 (2023)	UDK 62
UNIVERSITY OF BELGRADE - FACULTY OF MINING AND GEOLOGY	ISSN 03542904

UNDERGROUND MINING ENGINEERING - PODZEMNI RADOVI

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Publishing supported by: University of Belgrade - Faculty of Mining and Geology, Mining Section

Publisher: University of Belgrade - Faculty of Mining and Geology

For publisher: D.Sc. Biljana Abolmasov, Dean of Faculty of Mining and Geology

Printed by: SaTCIP, Vrnjačka Banja

Circulation: 200 copies

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UNDERGROUND MINING ENGINEERING 42 (2023)	UDK 62
UNIVERSITY OF BELGRADE - FACULTY OF MINING AND GEOLOGY	ISSN 03542904

The first issue of the journal "Podzemni radovi" (Underground Mining Engineering) was published back in 1982. Its founders were: Business Association Rudis - Trbovlje and the Faculty of Mining and Geology Belgrade. After publishing only four issues, however, the publication of the journal ceased in the same year.

Ten years later, in 1992, on the initiative of the Chair for the Construction of Underground Roadways, the Faculty of mining and Geology as the publisher, has launched journal "Podzemni radovi". The initial concept of the journal was, primarily, to enable that experts in the field of underground works and disciplines directly connected with those activities get information and present their experiences and suggestions for solution of various problems in this scientific field.

Development of science and technique requires even larger multi-disciplinarity of underground works, but also of the entire mining as industrial sector as well. This has also determined the change in editorial policy of the journal. Today, papers in all fields of mining are published in the "Underground Mining Engineering", fields that are not so strictly in connection with underground works, such as: surface mining, mine surveying, mineral processing, mining machinery, environmental protection and safety at work, oil and gas engineering and many others.

Extended themes covered by this journal have resulted in higher quality of published papers, which have considerably added to the mining theory and practice in Serbia and which were very useful reading material for technical and scientific community.

A wish of editors is to extend themes being published in the "Underground Mining Engineering" even more and to include papers in the field of geology and other geosciences, but also in the field of other scientific and technical disciplines having direct or indirect application in mining.

The journal "Underground Mining Engineering" is published twice a year, in English language. Papers are subject to review.

This information represents the invitation for cooperation to all of those who have the need to publish their scientific, technical or research results in the field of mining, but also in the field of geology and other related scientific and technical disciplines having their application in mining.

Editors

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UDK 62 ISSN 03542904

Original scientific paper

EVALUATION OF IMMISCIBLE CO2 INJECTION IN HIGH WATER PRODUCTION RESERVOIR IN THE PANNONIAN BASIN

Ivan Al-Jeboore¹, Danica Milićević¹, Bojan Martinović¹, Milica Ješić¹

Received: June 02, 2023

Accepted: June 09, 2023

Abstract: Carbon dioxide (CO₂) flooding is one of the most important and most used enhanced oil recovery (EOR) method because it does not only increase oil recovery efficiency but also is used as an underground CO₂ storage. It is considered a very complex method as it involves knowing the fluid phase behavior with different CO₂ concentrations. It should be noted that oil swelling (volume increase) with the dissolution of carbon dioxide has a significant effect on increase of oil recovery. When this occurs, a significant decrease in the viscosity of the oil is observed. In this study, a reservoir 3D simulation modeling approach was applied to evaluate immiscible and miscible CO₂ flooding in a high WC reservoir. To reduce simulation time, the PVT composition was grouped into 5 fluid components. The 3-parameter, Peng-Robinson Equation of State (EOS) was used to match PVT experimental data by using the Schlumberger's ECLIPSE PVTi software. One-dimensional slim-tube model was defined using ECLIPSE 300 software to determine the minimum miscibility pressure (MMP) for injection of CO2. Beside this approach, an analytical MMP estimation was carried out using several correlations. Schlumberger Petrel software was used to set up a 3D simulation model of a static and dynamic model. Various scenarios of immiscible and CO₂ injection have been simulated using ECLIPSE 300 software and these results have been compared.

Keywords: CO₂, Enhance Oil Recovery, Miscible fluids

1 INTRODUCTION

The use of carbon dioxide flooding to increase oil recovery started in 1950 and has been very successful. This success is firmly based on many laboratory studies, field trials and application experiences. Carbon dioxide is highly soluble in oil and soluble, to a lesser extent, in water. At the same time uses the following properties are known which enable carbon dioxide use in enhanced oil recovery when carbon dioxide mixes with the reservoir liquids:

- decrease in viscosity of crude oil and increase in viscosity of water.

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- oil swelling and oil density reduction.
- multiple contact miscibility with hydrocarbons.
- acidic type interaction with the formation carbonates and clays.

As CO_2 is much more soluble in water than hydrocarbon gases, it increases the water viscosity which leads to better sweep efficiency. Also, when carbon dioxide is dissolved in water some carbonic acid is formed. The acid etches carbonates and clays. This etching opens and widens throats between formation grains and the permeability of carbonate rocks increases by 675%, and sandstone rocks by 515%. The acidic environment also reduces swelling of clays. These have a significant effect on increasing reservoir permeability.

Carbon dioxide could displace oil by either miscible or immiscible displacement. For pressures below MMP, immiscible displacement of oil takes place, in which oil viscosity reduction, swelling of reservoir oil, reduction of interfacial tension, and solution gas drive are major driving mechanisms. This combination of mechanism enables a portion of the reservoir's remaining oil to be mobilized and produced. At pressures above MMP, the most dominant mechanism is miscibility between CO₂ and the reservoir oil. Miscible displacement by CO_2 is a much-preferred process to immiscible displacement. The miscible process may apply to heavy oils (ECLIPSE, 2014). In this study our main aim is to try to achieve a first contact miscibility to approach a zero interfacial tension and eventually increase a capillary number to infinity (Eq 1) as it is presented in Figure 1.



Figure 1 Capillary number behavior (Lake L., 1989)

On the other hand, CO_2 injection can have some disadvantages compared to other injection processes. One of the main problems in achieving profitable CO_2 flooding has been the high mobility of the CO_2 . The relatively low density and viscosity of CO_2 compared to reservoir oil are responsible for gravity tonguing and viscous fingering. This behavior was observed on several sandstone oilfields where CO_2 projects have been ongoing for almost 4 years. The main conclusion from these projects is that CO_2 fingering in Pannonian sandstone reservoirs is very hard to control due to different relative permeabilities which leads to poor sweep efficiency. This problem can be solved by introducing a WAG injection where water is responsible for controlling the CO_2 saturation front.

2 PRODUCTION HISTORY

XX reservoir started production in 1968. The first stage of production was relatively fast, where 8 wells were drilled in the first year of production which led to a stable oil production rate with a very uniform WC increase in wells. In 2005, a significant production intensification was made which led to noticeable increases in water production. Introducing new production wells, this trend has continued and rapidly increase from 1500 m³/month to 2000 m³/month of oil (Figure 2).

Even though several actions were taken to reduce the amount of water production to some previous levels (after intensification), it was not achieved. Pressure data suggests that the reservoir is under a heavy waterdrive mechanism, which is later confirmed by a material balance calculation. This means that to achieve a first contact miscibility, we need to inject less amount of CO_2 in comparison with no aquifer mechanism. As this reservoir is among the best reservoirs with the highest values of permeabilities (core data in some wells have permeability more than 1000mD), the voidage replacement ration is almost 1. Considering all previously mentioned facts, this reservoir was considered among the first priorities for EOR study.



Figure 2 Reservoir and production history

3 SCREENING CRITERIA

The initial step before any Enhanced Oil Recovery method to a field (or more commonly to the specific reservoir or even oil containing strata) is to decide which oil production enhancement method or methods are most appropriate. This is done based on reservoir and oil properties analysis so named screening step. Screening studies are designed to assess the feasibility of using the enhanced oil recovery method based on a limited number of reservoir and fluid properties. Those properties are mostly regarded as critical. Screening is a comparison of the averaged characteristics of the reservoir with tabulated criteria of various methods applicability. The set of tabulated criteria is made based on the joint international oil industry experience. Taking into account reservoir and fluid description presented in Figure 3, a screening criteria was done as illustrated in Figure 3 and Figure 4. The screening criteria mentioned here are meant to be used as a first order of screening, and more detailed studies must be undertaken before a decision to implement new CO₂ flood projects. Reservoir screening criteria inputs are presented in Table 1.

Table 1 Reservoir properties for EOR screening

Table I Reservoir properties for EOR screening	
Initial reservoir pressure (bar)	148.3
Reservoir temperature (°C)	81
Oil viscosity (cP)	1.39
Formation type	Sandstone
Average permeability (mD)	1000
Oil density at standard conditions (kg/m^3)	855

Properties	Nitrogen and flue gas	Hydrocarbon	Carbon Dioxide	Immiscible Gases	Miscellar/polymer, ASP, and alkaline flooding	Polymer flooding	Combustion	Steam
Oil API Gravity	> 35 Average 48	> 23 Average 41	> 22 Average 36	> 12	> 20 Average 35	> 15, < 40	> 10 Average 16	> 8 to 13.5 Average 13.5
Oil Viscosity (cp)	< 0.4 Average 0.2	< 3 Average 0.5	< 10 Average 1.5	< 600	< 35 Average 13	>10, <150	< 5,000 Average 1200	< 200,000 Average 4,700
Composition	High % C1-C7	High % C2-C7	High % C5-C12	Not critical	Light, intermediate. Some organic acids for alkaline floods	Not critical	Some asphaltic components	Not critical
Oil Saturation (PV fraction)	> 0.40 Average 0.75	> 0.30 Average 0.80	> 0.20 Average 0.55	> 0.35 Average 0.70	> 0.35 Average 0.53	> 0.70 Average 0.80	> 0.50 Average 0.72	> 0.40 Average 0.66
Formation Type	Sandstone or Carbonate	Sandstone or Carbonate	Sandstone or Carbonate	Not critical	Sandstone preferred	Sandstone preferred	High porosity sandstone	High porosity sandstone
Net Thickness (ft)	Thin unless dipping	Thin unless dipping	Wide range	Not critical if dipping	Not critical	Not critical	> 10 feet	> 20 feet
Average Permeability (md)	Not critical	Not critical	Not critical	Not critical	> 10 md Average 450 md	> 10 md Average 800 md	> 50 md	> 200 md
Depth (ft)	> 6000	> 4000	> 2500	> 1800	< 9000 Average 3250	< 9000	< 11500 Average 3500	< 4500
Temperature (deg F)	Not critical	Not critical	Not critical	Not critical	< 200	< 200	> 100	Not critical

Figure 3 Results of screening criteria for XX reservoir



Figure 4 Spider plot of screening result for XX reservoir

Reservoirs suitable for EOR by implementing CO_2 floods have various degrees of suitability depending on the intrinsic reservoir and oil properties. The range of reservoir characteristics and fluid properties suitable for CO_2 miscible injection is quite broad, and ideal reservoirs should have:

- Oil density <900 kg/m3
- Oil saturation > 25%
- Reservoir pressure > 0.9 MMP
- Porosity >15%
- Permeability > 1mD
- Moderate Heterogeneity

All these parameters were analyzed in detail and presented in the next sections.

4 PVT DATA AND FLUID CHARACTERIZATION USING EQUATION OF STATE

Reservoir fluid and injection gas behavior under different pressures and injection concentrations are essential for accurate project performance prediction. Complex reservoir fluids behavior is understood based on laboratory experimental data and thermodynamic modeling. All this undertaking allows to predict fluid behavior in the feasible range of Pressure, Volume and Temperature and to see at which PVT conditions the fluid will be a single phase or will separate into multiple components. This is especially important as simulation flowing equation, or Darcy's flow rate equation, are only applicable to the single-phase flow. Multiphase flow is described by much more complex equations than Darcy's one.

Several PVT analyses were conducted in XX reservoir during the early period of production. The analysis covered the main PVT experiments.

- Constant composition expansion
- Differential liberation
- Viscosity test

Based on available information regarding PVT data and sampling pressures, two samples were neglected as they were not representative. It was decided to proceed with PVT laboratory data from well XX-9 as they are the most accurate. Laboratory PVT data are presented in Table 2, Table 3 and Table 4.

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Step	Pressure (at)	Pressure (bar)	$\Delta V (cm^3)$	ΔV *100/Vo (%)	Relative volume
1	300	305.0	0	0	0.9851
2	275	279.7	0.15	0.2034	0.9871
3	250	254.3	0.31	0.4203	0.9892
4	225	229.0	0.485	0.6576	0.9916
5	200	203.7	0.666	0.903	0.994
6	175	178.3	0.865	1.1729	0.9966
7	150	153.0	1.066	1.4454	0.9993
8	145	147.9	1.116	1.5132	1.0000
9	143	145.9	1.396	1.8929	1.0037
10	142	144.9	1.915	2.5966	1.0107
11	137	139.8	2.392	3.2434	1.017

Table 3 Differential liberation experiment - sample from well XX-9

Step	Pressure (at)	Pressure (bar)	Rs (m ³ /m ³)	Bo (rm ³ /sm ³)	Oil density (kg/m ³)	Oil viscosity (cP)
15	300	303.98	52.92	1.1518	780.0	1.65
14	275	278.64	52.92	1.1543	778.3	1.60
13	250	253.31	52.92	1.1571	776.5	1.55
12	225	227.98	52.92	1.1599	774.5	1.50
11	200	202.65	52.92	1.1631	772.5	1.45
10	175	177.32	52.92	1.1664	770.2	1.40
9	150	151.99	52.92	1.1697	768.0	1.34
8	145	146.92	52.99	1.1704	767.6	1.39
7	125	126.66	45.54	1.1535	774.0	1.45
6	100	101.33	37.11	1.1356	780.6	1.69
5	75	75.99	27.69	1.1149	788.6	1.80
4	50	50.66	18.38	1.0949	796.7	2.05
3	25	25.33	9.39	1.0761	804.2	2.33
2	0	0.00	0.00	1.0531	814.2	2.62

Since this reservoir started to produce in 1969, no equipment was available at the time for determining the fluid composition. As liquid phase (oil) composition was not determined by chromatography, a simplified approach was applied to obtain a representative monophasic sample for EOS tuning. Using the only available gas composition it was assumed that the liquid composition is strictly 100% C7 fraction. The

calculation of the reservoir fluid sample was done by recombination method using available GOR at separator conditions and reservoir oil density according to the equations 2 and 3.

$$z_i = \frac{2130 \cdot \rho_o \cdot x_i + M_o \cdot GOR \cdot y_i}{2130 \cdot \rho_o + M_o \cdot GOR}$$
(Eq. 2)

Where:

- M_o molecular weight of the separator liquid sample
- ρ_o density of separator liquid sample at separator pressure and
- temperature, lb/ft³
- GOR recombined gas-oil ratio, scf/bbl
- x_i mole fraction of component i in the separator liquid phase sample
- y_i mole fraction of component i in the separator gas phase sample

Reservoir oil molar weight was calculated using oil density at separator conditions according to the following expression:

$$M_o = 0.048923 \cdot e^{(9.88378 \cdot \gamma)} - 33.085468 \cdot \gamma + \frac{39.598437}{\gamma}$$
(Eq. 3)

The calculated reservoir composition is presented in Table 4.

Component	Gas composition	Oil composition	Reservoir fluid
Component	Component (%)		(%)
N2	2.69	0	1.0358
CO2	1.05	0	0.4043
C1	88.15	0	33.942
C2	2.16	0	0.83171
C3	1.53	0	0.58913
IC4	0.87	0	0.33499
NC4	1.12	0	0.43126
IC5	0.66	0	0.25413
NC5	0.7	0	0.26954
C6+	1.07	100	61.907

Table 4 Recombined fluid composition.

In this study, Schlumberger software PVTi was used for characterization of the reservoir oil sample. Insufficient description of heavier hydrocarbons reduces the accuracy of PVT predictions. Therefore, it is necessary to split the plus components into two or three pseudo components, specifically when there are many of them compared to the other components. As shown in Table 4, 61.907% of fluid is C6+.The C6+ component was split into three pseudo components by Whitson's method as shown in Table 5. Critical

properties Tc and Pc as well as acentric factor ω for the new pseudo components, were defined by Lee–Kesler correlation.

Reservoir Fluid	Gas	Oil	Reservoir Fluid	Splitting	Group	P.Comp
CO2	2.69	0	0.40	CO_2	1	CO_2
N2	1.05	0	1.04	N2	2	C1-N2
C1	88.15	0	33.94	C1	2	CI-IN2
C2	2.16	0	0.83	C2	3	$C^{2}C^{2}$
C3	1.53	0	0.59	C3	3	C2-C3
IC4	0.87	0	0.33	IC4	4	
NC4	1.12	0	0.43	NC4	4	$C_{1}C_{5}$
IC5	0.66	0	0.25	IC5	4	C4-C5
NC5	0.7	0	0.27	NC5	4	
				FR1	5	
C6+	1.07	100	61.91	FR2	5	C6+
				FR3	5	

 Table 5 Fluid composition after splitting and grouping

The main reason for grouping components is to speed-up the compositional simulation. In a compositional simulation the number of grouped components depends on the process that is modeled. For miscibility, more than 10 components may sometimes be needed. In general, 4-10 components should be enough to describe the phase behavior (ECLIPSE, 2014). The main issue for grouping is to collect components with similar molecular weights. For example, group C7 with C8 rather than with C2. As one would expect, the properties of C7 and C8 are similar, while the properties of C7 and C2 are very) different. Obvious candidates are to group iC4 with nC4 and to group iC5 with nC5. An exception to this rule is that N2 is added to C1, and CO_2 is usually added to C2. As the purpose of this study is to evaluate CO_2 injection potential, CO_2 must be single component in order to be pre-defined in ECLIPSE 300 using the keyword STREAM. The last stage was to fit an EOS to have an agreement between the observed data and the results calculated with the EOS. The 3-parameter, Peng-Robinson Equation of State (EOS) was used in this paper. Peng–Robinson, a cubic EOS that was developed by Peng and Robinson in 1976 (Ding-Yu et al., 1976), has been shown to accurately model hydrocarbons and is the most widely used EOS in compositional reservoir simulators (Søreide & Whitson, 1992). Tuning results are presented in Figure 5.



Figure 5 PVT experiment matching results

5 DETERMINING THE MINIMUM MISCIBILITY PRESSURE (MMP)

The minimum miscibility pressure (MMP) is the pressure corresponding to the transition point to the maximum achievable recovery (Figure 6).



Figure 6 Minimal miscible pressure determination

The pressure of complete miscibility of carbon dioxide and the oil is determined experimentally. There are few methods to do it, but the most common are: gravitational-stable and slim tube tests. As there was not any slim tube experiment, MMP was calculated empirically using available correlation, one dimensional model to simulate a slim tube experiment, and already matched PVT model using 3 parameter PR EOS. Empirical correlations showed that based on our C5+ concentration the MMP is in range of 153 - 260 bar (Table 5). In Table 6 MMP calculations using empirical correlation is presented.

Table 6 MMP calculation using empirical correlation

Author	Calculated MMP (bar)
Cronquist (Cronquist C., 1978)	215.68
Lee (Lee I., 1979)	181.62
Yelling-Metcalfe (Yelling W. et al., 1980)	153.02
Orr-Jensen (Orr F. et al., 1987)	145.49
Glaso (Glaso O., 1985)	170.64
Alston (Alston R.et al., 1985)	260.91
Emera-Sarma (Emera M. et al., 2006)	240.9
Shokir (Shokir E., 2007)	207.84
Chen (Chen B. et al., 2013)	241.09

The matched PVT model gave much higher values comparing to empirical correlation presented above (Figure 7).

Expt FCMP1 : First Contac	ct Miscibility Calcula	tion
Peng-Robinson (3-Par Lohrenz-Bray-Clark Viscosity		PR corr.
Specified temperature Injection gas	Deg C CO2	83.0000
First contact miscibility p	ressure BARSA	371.2892

Figure 7 MMP estimation based of PR EOS

One-dimensional compositional simulation of the slim-tube model was performed to determine the minimum miscibility pressure (MMP) of CO₂ with the reservoir fluid. The ECLIPSE 300 was used. As it can be seen from the Figure 8, the MMP is estimated to be 100bar which is significantly lower than MMP obtained by EOS and empirical correlations. Due to huge differences in MMP values, this study was done using the already matched PVT model as it is most reliable, where MMP is estimated to be 371.29 bar. Even though the reservoir pressure drop from initial values was very small, it is still much below the MMP value that was calculated. On the other hand, the fracture pressure was estimated using hydraulic fracture pressure data from already fractured wells in the nearby fields and that value is estimated to be 210 bar. That suggests that it is not possible



to achieve first contact miscibility (FCM) in the reservoir as the bottom hole pressure must be set below that value.

Figure 8 MMP estimation based on one dimensional model

6 MODEL ADAPTATION AND PREDICTION ANALYSIS

Before starting any prediction analysis, it is required to history match the reservoir production and pressure to obtain current fluid saturations and pressure. This process was done using all available information gathered from the beginning of production. The history matching process was carried out using ECLPISE 300 compositional simulator with adapted PVT data. A relatively good match was achieved by using assisted history matching tool with changing fluid relative permeabilities end points. Matching results are presented in Figure 9.

Evaluation of immiscible CO₂ injection in high water production ...



Figure 9 History match results for reservoir XX

It must be noted that the water production mismatch in the late period of production is directly related to the inaccurate fluid measurement during commingled production which was carried out on several wells on that field. The oil production rate and oil production cumulative were matched with error of $\pm 5\%$. The remaining oil saturation based on history matching results is concentrated in the central part of the reservoir as it can be seen from Figure 10.



Figure 10 Remaining oil saturation at the end of HM

After the history matching process, the model is set for prediction of different scenarios. Several injection scenarios were analyzed which take into consideration the amount of injected CO_2 gas.

As hydrocarbon components exist in the oil and gas phases they are not allowed to dissolve in the aqueous phase. Usually, this assumption is adequate since the hydrocarbon solubility in water is low over the range of temperature and pressure for gas injection. CO_2 , however, is an exception to this assumption. The solubility of CO_2 in water is much higher than that of hydrocarbon components and is a factor that cannot be neglected in the simulation process. To model a process where CO_2 is soluble in aqueous phase, in Schlumberger ECLIPSE E300 *CO2SOL* must be applies. The amount of CO_2 dissolved in water, and other aqueous phase properties, are computed using solubility data that is entered with either the *SOLUBILI*, *SOLUBILS* or *SOLUBILT* keywords. Due to lack of laboratory data of CO_2 solubility at different injection pressures, an analytical model was developed using reservoir water salinity at different reservoir pressure steps. Based on Chang method (Chang et al., 1996), properties of water are calculated for different pressures at reservoir temperature and given in Table 7. As can be seen from the table, water viscosity is considered not to be affected by CO_2 injection above 50°C temperature and therefore it remains constant.

Table 7 Water properties with dissolved CO₂

P (bar)	R_{sw} (sm ³ /sm ³)	B _w (rm ³ /sm ³)	μ _W (cP)	C_w (bar ⁻¹)
100	3,7	1,0277	0,3440	5,34E-05
120	4,0	1,0267	0,3440	5,39E-05
140	4,3	1,0258	0,3440	5,43E-05
160	4,7	1,0248	0,3440	5,47E-05
180	5,0	1,0239	0,3440	5,51E-05
200	5,3	1,0229	0,3440	5,54E-05
220	5,6	1,0219	0,3440	5,56E-05
240	6,0	1,0210	0,3440	5,58E-05
260	6,3	1,0200	0,3440	5,60E-05

Different scenarios were run with daily injection rates from 100.000 m³/d to 500.000 m³/d. The CO₂ injection rate was assigned to 3 wells located in the central part of the reservoir. Simulation results are presented in Table 8 and in Figure 11.

		ater production

	W. 11 sheet 'm	Injection v	DE	
Scenario	Well shut in conditions	Injection parameters	Number of wells	RF (%)
Scenario I		Qinj=100.000 m ³ /d BHIP (max)=200 bar		70.68
Scenario II		Qinj=200.000 m ³ /d BHIP (max)=200 bar		71.5
Scenario III	Qo _{min} =0.5m ³ /d WC=99%	Qinj=300.000 m ³ /d BHIP (max)=200 bar	3	72.33
Scenario IV		Qinj=400.000 m ³ /d BHIP (max)=200 bar		72.86
Scenario IV		Qinj=500.000 m ³ /d BHIP (max)=200 bar		73.09



Figure 11 Simulation results for different injection scenarios

7 CONCLUSION

Estimating MMP using slim tube test is essential for CO_2 injection study as different approaches give a very wide range value of MMP.

Reservoir fluid characterization is considered as the most important parameter when planning CO₂ EOR study. A precise determination of C7+ critical values (Tc, Pc) and acentric factor ω has a significant impact on the minimum miscibility pressure.

The injection rate is the most important parameter that can affect the oil recovery factor, specifically in highly permeable reservoirs.

In the immiscible CO_2 injection, increasing the gas injection rate leads to faster movement of CO_2 front toward production wells which resulted in increase in CO_2 content and well GOR, thus it causes the shutdown of some of the production wells and the oil recovery factor will be less.

The amount of CO_2 solubility in aqueous phase has a large impact of the final recovery factor especially in highly water cut reservoir. A significant amount of CO_2 is soluble in aqueous phase, and this is causing a high CO_2 concentration on nearby production wells.

An interesting finding from the literature study is that an oilfield that has behaved well under waterdrive mechanism seems to behave well under CO_2 flooding. Another finding

is that increased oil production, up to a certain point, is almost linear to the amount of CO_2 injected.

As this study did not cover reservoir fracture pressures, it is highly recommended to estimate this parameter and use it as a constrain, (otherwise simulator will increase bottom hole injection pressure to abnormal values to achieve given injection rates and oil RF can be unreal).

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UNDERGROUND MINING ENGINEERING 42 (2023) 19-25 UNIVERSITY OF BELGRADE - FACULTY OF MINING AND GEOLOGY UDK 62 ISSN 03542904

Review paper

INFLUENCE OF TYPE AND QUANTITY OF FILLER ON CHANGE OF DENSITY OF SULFUR CONCRETE

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Received: March 25, 2023

Accepted: April 26, 2023

Abstract: Concrete is a material that has been used for centuries and is often modified using polymers. In the last fifty years, synthetic polymers have been used for the modification of concrete, but also for the production of concrete. In recent decades, sulfur concrete has been an interesting product that can be used mainly in low-rise construction due to its characteristics. In this work, we used the starting mixture for the preparation of sulfur concrete (sand, elemental sulfur with the addition of modified sulfur and fillers) heated to a temperature of 120 °C to 170 °C and homogenized. The results of previous research on the production of sulfur concrete showed that the density of the obtained product changes depending on the type as well as the amount of filler added to the basic mixture based on raw materials. Talc, microsilicon, plate alumina and fly ash were used as fillers. The amounts of fillers were 0%, 1%, 3%, 5%, 7% and 10%.

Keywords: Filler, Density, Sulfur concrete

1 INTRODUCTION

Sulfur concrete is a relatively new material. In its final form, it is similar to cement concrete, but its production, use and testing are significantly different (ACI, 1998). It is a thermoplastic material obtained by mixing modified elemental sulfur and mineral aggregate at temperatures above the melting temperature of sulfur (120-130 °C). By cooling below those temperatures, sulfur concrete hardens very quickly, acquiring the expected physical and mechanical characteristics (Blight et. al, 1978). As with other concrete materials, the characteristics of sulfur concrete depend significantly on the type and shape of the aggregate, the sulfur modifier and the applied proportions of mineral aggregate and binder (Jordan et. al, 1978; ACI, 2004, Vroom, 1977).

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The aim of all previous research (Vlahović et. al, 2006; Boljanac et. al, 2006; Vidojković et. al, 2006) in the field of development of the production and application of sulfur concrete was to make a simple and clear formulation, with high-quality physical, mechanical, chemical, and ecological characteristics of the obtained product. This product would enable the wide application of sulfur concrete, and thus significant consumption of elementary sulfur, which is a secondary product of oil production. For the widest possible application of sulfur concrete, it is necessary to ensure appropriate resistance to various mechanical and chemical influences (ACI, 2004).

The combination of sulfur with a mineral aggregate of the appropriate granulometric composition fast and quality connects the elements of the aggregate, resulting in a very strong and chemically resistant material. Over time, recrystallization of sulfur and changes in crystal modification occurs in such material, and the result of such a process is the weakening of the original characteristics of sulfur concrete (Vroom, 1977). This phenomenon significantly shortens the life of sulfur concrete, which results with limits its application, so later research required finding a solution that will suppress or completely prevent the change of crystalline modification of sulfur and appropriate additives are known, which ensure a significantly longer service life of sulfur concrete (20-30%), in which it retains its original physical-mechanical and chemical characteristics (Vroom, 1981; Sodeberg et. al, 2001). Such combinations are commonly called modified sulfur or sulfur cement.

Numerous studies were conducted with the aim of defining the optimal combination of mineral aggregates which, together with modified sulfur, will provide the necessary, high-quality results. Ash and other waste materials were used as a binder in many researches, because the practical application of such technological solutions reduces the cost of binder materials to a minimum and at least partially solves two environmental problems (William & Liao, 1981; Vidojković et. al, 2006).

The main advantage of sulfur concrete is the possibility of its use as a highly resistant construction material in industrial plants and in other locations where an acidic environment or an environment saturated with various salts prevails. Cement concrete in such locations corrodes and deteriorates in a very short period, which leads to the conclusion that its application is practically impossible. In these places of application, the service life of sulfur concrete far exceeds the service life of all other known construction materials (McBee et. al, 1986). Another specific characteristic and advantage compared to cement concrete is the short setting time and the achievement of a high level of compressive strength. Since it achieves its maximum mechanical characteristics in less than 24 hours, the period until pouring sulfur concrete is put into operation is much shorter than in the case of cement concrete (ASTM C, 1997; ASTM C 2003).

Sulfur concrete has numerous useful characteristics that can give it an advantage over cement concrete (Khademi & Kala, 2015). Tensile, compressive and bending forces, as well as the fatigue life of the material, are better than cement concrete. Sulfur concrete has exceptional resistance to the influence of many acids and salts, even when they are in very high concentrations. It hardens very quickly and achieves a minimum of 70-80% of the required characteristics already within 24 hours. It can be produced and installed throughout the year in all weather conditions, even at temperatures below 0 °C. Due to the hydrophobicity of sulfur, it has very low water permeability and can be used as a waterproofing material.

It has been shown (ACI, 1998) that certain types of fillers with grain size below 100 µm have a positive effect on the physical and mechanical characteristics of sulfur concrete. By filling the free space between individual grains of larger aggregate, they reduce the porosity of the concrete, increase the overall density of the concrete mass, and thus the resistance of the concrete to mechanical influences increases. The aim of this work was to investigate the influence of several types of fillers on the density of sulfur concrete.

2 EXPERIMENTAL PROCEDURE

In all experiments, river sand (grain size up to 2mm), elemental sulfur (obtained as a byproduct in the oil refining process of the Pančevo oil refinery) was used as an aggregate during the preparation of sulfur concrete, with the addition of modified sulfur, while talc, microsilica, sheet alumina and fly ash of the Nikola Tesla Power Plant "A" in Obrenovac. The amount of filler was 1%, 3%, 5%, 7% or 10%, while the amount of sand was successively reduced and amounted to 69%, 67%, 65%, 63% and 60% in relation to the total amount of the mixture. The sulfur content in all mixtures was constant and amounted to 30%. Sulfur concrete without fillers was prepared as a reference sample.

Sand with the addition of filler is placed in open reactors in laboratory conditions and heated to a temperature of 120 to 140°C. Melted sulfur in amounts of 30% was added to this mixture with intensive and continuous mixing. At the achieved temperatures, the sulfur in such a molten state coated the sand particles and the filler was homogenized. After homogenization, the obtained product was poured into special molds with intense vibration of the substrate for better settlement of the obtained mixture. The concrete in the molds cooled spontaneously to room temperature, after which the density of the samples was measured.

3 RESULTS

The test results showed that the amount of filler added to the mixture has a significant effect on the density of the obtained product. The density of the reference sample, which was prepared from sand and sulfur in a ratio of 70:30 without the addition of filler, was





Figure 1 Changes in the density of sulfur concrete as a function of the type and amount of filler

Using ash as a filler, the density of product did not have significantly changes, with the fact that the density dependence curve on the amount of filler shows a slight maximum for the addition of 3% and 5% ash (2,180 g/cm³). With the addition of 7% and 10% ash, the value concrete density decreased to slightly lower values than the initial sample (2,140 and 2,150 g/cm³). During the homogenization of the initial mixture at temperatures above 130 °C, it was observed that after the addition of molten sulfur, the sample was visually wet, and after a few minutes of mixing, it almost dried up, and the homogenization process itself was difficult. This phenomenon occurs due to the effect of wetting the ash particles with sulfur, whereby the sulfur coats the ash particles and binds to it with adhesive forces. This reduces the role of sulfur in the basic mixture for obtaining concrete, which at high temperatures has the function of enabling a homogeneous and dense binding of sand in a two-component system, thus achieving adequate conditions for obtaining concrete of higher strength. By visual analysis of the obtained sample of concrete with ash, with all individual compositions of ash, significant voids were observed, which can significantly affect the compactness and quality of sulfur concrete as a final product.

With the addition of plate alumina as a filler, it was shown that the density of the obtained concrete decreased to 2.116 g/cm^3 with the addition of 1% alumina, after which the density values increased sharply as a function of the increase in the added filler, and reached a value of 2.459 g/cm^3 for the sample with 10% filler, and these are practically

the highest concrete density values obtained in these studies. This data points to the assumption that the addition of alumina enables uniform homogenization of the components that make up sulfur concrete, while the shape of the density dependence curve on the amount of filler shows that with further addition of filler, the maximum density of the sample would be reached (the curve tends to the asymptote), after which further addition fillers would not change the density value.

The results of the change in concrete density depending on the amount of added filler in the case of microsilica show slightly different results. The density compared to the reference sample without filler jumps sharply with the addition of 1% microsilica (2.286 g/cm³), which achieves the maximum density of the final product with this type of filler. With the addition of 3% microsilica, the density of concrete is slightly lower (2.262 g/cm³), but with the addition of 5% microsilica, the value of concrete density drops sharply to 2.111 g/cm³. By further increasing the amount of this filler, the density of the concrete still tends to stagnate, so that with 10% microsilica, the value of the density of the obtained concrete finally drops to 2.045 g/cm³. A photo of sulfur concrete samples with 5% filler is shown in Figure 2.



Figure 2 Photographs of samples of sulfur concrete with 5% filler (A5alumina, S5-microsilica, P5-fly ash and T5-talc)

The increase in the amount of talc as a filler in the production of sulfur concrete shows an increase in the density of concrete with an increase in the amount of talc up to 5% (concrete density 2.421 g/cm^3), after which, with a further increase in the amount of talc, the density of concrete decreases to 2.333 g/cm^3 for 10% talc. When mixing the basic components, it was observed that increasing the amount of talc facilitates the

homogenization of the sample and the pouring of the sample into the appropriate molds for cooling and the formation of sulfur concrete.

4 CONCLUSION

Examinations of changes in the density of sulfur concrete depending on the type and amount of filler showed that the density of concrete increases with the increase in the amount of added filler (except in the case of flat alumina, where with the addition of 1% alumina, the concrete density value decreased compared to the reference sample, so that with a further increase in the amount of filler, the concrete density increased). The highest concrete densities were achieved with 1% microsilica (2.286 g/cm³), 5% talc (2.421 g/cm³) and 10% plate alumina (2.459 g/cm³), while with the addition of ash of 3% and 5% the value was achieved concrete density of 2,180 g/cm³.

Since the density of concrete is an important factor affecting the physical characteristics of the product, these tests show that talc and microsilica as fillers showed better properties than plate alumina and fly ash.

Acknowledgement

This work was financially supported by the Ministry of Science, Technological Development and Innovation of the Republic of Serbia (Grant Nos. 451-03-47/2023-01/200026).

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UNDERGROUND MINING ENGINEERING 42 (2023) 27-36 UNIVERSITY OF BELGRADE - FACULTY OF MINING AND GEOLOGY UDK 62 ISSN 03542904

Review paper

LEGAL FRAMEWORK FOR RECULTIVATION OF DEGRADED AREAS CAUSED BY MINING EXPLOITATION

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Received: May 22, 2023

Accepted: June 10, 2023

Abstract: During the exploitation of coal, both surface and underground exploitation systems damage larger or smaller areas of land. Experiences show that land damage is significantly greater with the surface exploitation system, where the content of the space is degraded not only in the contour of the mine and the surrounding area, but changes are made in the natural flows and conditions as well.

The article presents the results of a study of the regulatory framework the issues of reclamation in some world countries also in Serbia. Practical experience of reclamation has shown that utilisation and devastation by mining activities present not only danger to affected areas but also an opportunity to correct negative developments and to plan in accordance with the expectations of the population and respected to laws.

Keywords: recultivation, reclamation, coal mining, legal regulation

1 INTRODUCTION

Recultivation can produce more value for the environment and create a better situation for post-mining land. Recultivation obligation policy is a great way to introduce a restorative approach to the environment, but main problem is the inconsistency of the mining companies for their reclamation obligation. It is very importatant conceptualizing the strengthening of the recultivation obligation policy in the Mining Act based on the understanding of preventing environmental damage and also for humans.

Mineral and coal mining often increases soil density, erosion, sedimentation, landslides, disruption of flora and fauna, and disruption of public health, which leads to microclimate change (Goudie and Viles, 2013). The post-mining impacts have resulted in land morphology, topography, and landscape changes. Landscapes on the post-mining areas are usually irregular, causing steep holes and tumps), after which, the land becomes

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unproductive. Mining activities have a high risk of environmental damage and create environmental justice issues, which have become a global concern (Preston, 2018). Reclamation forces mining companies to restore the caused environmental losses. Land reclamation aims to repair the damage to post-mining ecosystems by improving soil fertility and planting lands at the surface (Listiani at all, 2023). Land recultivation seeks to make the land more productive and create a far better situation for environment after coal mining. Moreover, a good recultivation policy is a great way to introduce a restorative approach to the environment plus the sustainable management of reclaimed land. Mining companies should conduct reclamation of unused post-mining land inside and outside ex-mining zone. Land outside an ex-mining zone includes (1) landfill; (2) stockpiling of raw materials; (3) transportation roads; (4) plant/processing; (5) offices and housing. For the successfully recultivation plan and post-mining plan to the proposed changes).

After the completion of mining operations, enterprises must complete reclamation of the land within the specified period and government departments should organize timely acceptance after the completion of land reclamation. For agricultural land reclamation, especially after restoration of the original use of arable land, acceptance of the main content should include: reclaimed land area, surface cover thickness, ground slope and drainage function, the physical and chemical properties of the soil are in line with the standards of arable land, and the failure to meet the acceptance criteria, should not pass the acceptance (Guo at all, 2022). After the enterprise completes the land reclamation and the acceptance meets the standard, land should be established on a scientific basis, achieve a social balance between short-term and long-term interest and respect the interests of all parties.



Figure 1 Phases of land reclamation

2 TERMS - RECULTIVATION, RECLAMATION, RESTORATION, REVITALISATION

Although the term reclamation appeared at the beginning of the 20th century, the first attempts to rehabilitate degraded land date back to the end of the 19th century, note in Germany. The term "damaged land" means all those surfaces that have been affected directly or indirectly by a specific activity. The term devastated area is also usually used, which denotes a lifeless area after use, without a fertile soil layer suitable for future cultivation. Damaged land is brought to a new purpose through technical and biological reclamation, where land reclamation is a set of works for the rehabilitation (restoration) of productivity and economic value of damaged land, as well as improving the conditions of the surrounding environment.

Various terms have been used to describe the repair of land disturbed by mining and other inudtrial use of land use, including recultivation, reclamation, rehabilitation, reconstruction, renaturation, restoration and revegetation. In table 1 are shown basic definitition regarded to recultivation of degraded land.

Reclamation	represents restoring or giving the usable values to degraded land by appropriate land modelling, improvement of grounds properties, water regulation, soils restoration and roads construction.	
Rehabilitation	means the return to the natural state according to the original land development plan. The return to the natural state should be in accordance with the aesthetic qualities of the surrounding areas.	
Restoration	undestood as the return to the original state of the altered land, the state before degradation.	
Revitalization	is the term that covers both stages: reclamation and land development, and it means the state restoration, giving the opportunity to perform the utility function of this area.	

Table 1 Terms which are used to describe the remediation of degraded land

According to Jancura and Belachek (Jancura and Belachek, 2003), the newly crated landscape should satisfy the following requirements:

-"to be environmentally friendly;

- to harmonize with the natural environment, complementing the missing elements, increasing the number and variety of ecological niches;

- to meet aesthetic requirements;

- to meet the present and future needs of the local population and the region".
Considering the concept of recultivation, the analysis of researchers' views on the concept of "recultivation" shows that it is increasingly being replaced by the terms "revitalization", "renaturation", "restoration" or even "environmental remediation". Such understanding provides the harmonious inclusion of the restored landscape into the environment, taking into account landscape architecture (Ignatyeva, 2020).

Failure to plan and to start rehabilitation early in the life of the operation may create an obstacle to building the knowledge and capacity necessary to deliver a sustainable outcome that meets agreed success criteria (Australian Government 2016).

3 LEGAL FRAMEWORK IN WORLD

Mine reclamation is required by the governments of most countries. Before the mining process begins, companies develop mining plans that outline the process of land disturbance to obtain the mineral resource. The mining plan also outlines a reclamation procedure to restore the land to a productive post-mining land use (Skousen and Zipper, 2014) .This paper will give a brief overview of the legal regulations of some world countries China, USA, Australia, Germany, Poland.

CHINA

The State Council issued and implemented Land Reclamation Regulation (No.592 Order of the State Council of the People's Republic of China) On March 5, 2011. This LRR includes six chapters and 44 articles. That marks that China's land reclamation career entered into a new stage with cooperation of institution, standardization and legalization.

Compared with the "Stipulation on Land Reclamation" (SLR), which was issued in 1988, the main differences can be summarized by the following several aspects:

- 1. The definition of the reclamation object is more comprehensive. In addition to the damaged land by production and construction activities, damaged land by natural disasters is taken into consideration;
- 2. It has clarified the main responsibility of the land reclamation;
- 3. It has perfected the land reclamation obligations constraint mechanism;
- 4. the land reclamation incentive mechanism has been strengthened;
- 5. the department's responsibility is clarified further.

On December 11, 2012. based on LRR, Implementation Measures on Land Reclamation Regulation (IMLRR) was also publish and represent a detailed regulation on LRR. Regulation on Compiling Land Reclamation Plan (RCLRP) was issued in 2011, contains 7 parts (general rules, opencast coalmine, underground coal mine, metal mine, petroleum and natural gas (including coal-bed gas), construction projects, and uranium mine).

Further, Completion Standards on Land Reclamation Quality (CSLRQ) was issued in 2013 and are concentrated on the land reclamation quality.

USA

The first legislative act relating to the recultivation of disturbed land was adopted in 1939 (USA) when authors identified three stages in the evolution of legal support for recultivation. The first stage called the initial stage of the development of recultivation legislation (1939 to 1954). Second stage was the main period, when the basis for recultivation legislation was created is from 1955 to 1975. The third stage was devoted to the development of organizational and economic infrastructure.

AUSTRALIA

In Australia, for example, individual states and territories are responsible for mining, rehabilitation, and mine closure, including the adoption of individual laws, regulations, and guidelines. Industry and government agencies have established policies, principles, and guidelines for mine closure (Morrison-Saunders et al. 2016). Legislation on mine closure are either enacted as part of the mining sector or through specific environmental laws that apply to the mining companies. For instance, Environment Protection and Biodiversity Conservation Act (EPBC 1999) provides a uniform national framework for the environmental impact assessment of new projects. The largest share of control of the mining industry, including mine closure and recultivation, belongs to the Ministry of Minerals and Energy.

GERMANY

In Germany, according to the Federal Mining Act (BBergG) from 1980, mining activities do not end with the extraction of mineral resources. Rehabilitation represents an integral part of mining activity and mines operators are required under law to rehabilitate excavated sites. The surface affected mining must be rehabilitated as soon as the extraction work permits. Recultivation needs not necessarily mean restoring the land to its original state.

Practical experience of reclamation and restoration has shown that utilisation and devastation by mining activities present not only a burden or danger to affected areas but also an opportunity to correct negative developments and to plan in accordance with the expectations of the population and more freely than one could in a grown landscape (National Report).

POLAND

In Poland, recultivation is covered by 3 laws: Environmental Law, Geological and Mining Law and Act on the protection of agricultural and forest land. Undertaking the exploitation of mineral deposits is obliged to take the necessary measures to protect the resources of the deposit, the surface of the earth ..., successively carry out reclamation

of post-mining areas and restoration of other natural elements to the proper state (Environmental Law , art. 126).

4 LEGAL FRAMEWORK IN SERBIA

The policy of the state and local authorities regarding the protection of land from pollution and damage, as well as the mandatory undertaking of reclamation works, is regulated by several legal and by-laws, each of which, from its point of view, regulates this problem (Ivković at all, 2013).

In the field of mining, the basis is the Law on Mining and geological research, which orders that mining companies that have acquired exploitation rights are obliged to bring the land to a specific purpose during and after exploitation and carry out land reclamation activities in accordance with the reclamation project (Law on Mining and geological research, 101/2015,95/2018, 40/2021). The performance of mining exploitation is conditioned by the existence of prepared and approved investment-technical and project documentation, and the scope of recultivation and rehabilitation works is also determined by the valid regulations on the content of project documentation. The set of laws in the field of environmental protection also includes the protection of land from physical degradation and pollution by harmful materials. Only valid basic legal acts are listed here:

- Law on Environmental Protection,
- Law on Agricultural Land,
- Water law,
- Law on Forests,
- Law on Waste Management,
- Law on Environmental Impact Assessment,
- Law on Strategic Environmental Impact Assessment,
- Law on Integrated Prevention and Control of Environmental Pollution.

Legal and physical entity who use natural resources are obliged to keep up to date during the execution of works and performance of activities, as well as after their termination, plans and implements measures to prevent endangerment of the environment. Namely, whoever degrades the environment is obliged to carry out recultivation or rehabilitate the degraded environment in another way.

The assessment of the impact of the project on the environment is an integral part of the documentation without which the execution of the project cannot be started, and it is based on the preparation of the Study on the assessment of the impact on the environment (scope and content is determined by special regulations adopted on the basis of the above of the law).

5 COMPARATION LEGAL FRAMEWORKS IN SERBIA AND OTHER LEADING COUNTRIES

Recultivation of degraded areas caused by mining is under supervision of Ministry of Mining and Energy in Serbia, as well as regulatory function of state mining administration which are in the form of directorate, department or agency. The supervisory functions of state mining administration are defined in form of inspection and also belong to the relevant ministries. Advisory function is equally important as previous two described functions of state mining administration. In accordance with EU legislative, in Germany and Poland local entities has that role. Serbia is faced with lack of precisely defined aims of advisory service, but that does not mean that current units in state mining administration are not available for any kind of advice, in accordance with procedures. Table 2 shows relevant mining Ministries and their functions.

Country	The Competent Ministry	Regulatory function	Supervisory function	Advisory function
Serbia	Ministry of Mining and Energy		Mining Inspection	Not established
China	Ministry of Natural Resources (MNR)	Ministry of Environmental Protection	Inspection	Established
USA	Secretary for Economic Growth, Energy and Environment	The Bureau of Energy Resources (ENR)	Inspection	Established
Australia	Department od Mines, Industry and Safety	Regulated by states and territories	Inspection	Established
Germany	Federal Ministry for Economic Affairs and Energy	1 0	District and State Mining Authorities	Local entities
Poland	Ministry of Environment	District governors	District and State Mining Authorities	Local entities

Table 2 Relevant mining authorities and recultivation after degradation administration in countries

Source: Original

Based on the information exchange provided in Art. 21(3) of Extractive Waste Directive 2006/21/EC, the Best Available Techniques Reference Document for Management of Waste from Extractive Industries offers more than 700 pages of examples for good practice mining operations and best available techniques. Moreover, the Reference Document on BAT is not legally binding and the recommendation requires further specification, especially with regard to intact land reclamation after coal mining.Transposition of the Extractive Waste Directive 2006/21/EC into national laws, regulations and administrative provisions is is mandatory for EU members. The implementation EU Directive into national laws and administrative provisions is very different within Europe.

For example, in Germany there is one single legislative regime or framework - the national mining law, BBergG (1980) - bringing together all mining operations and the general environmental laws to speed up and simplify the complex planning process.

The law in Serbia defines that the Exploitation Holder is obliged to recultivate the land in accordance with the technical project of the technical and biological reclamation, which is an integral part of the main or supplementary mining project, and it is reported to the Ministry, i.e. the competent authority of the autonomous province and the ministry responsible for agriculture and water management, i.e. the ministry responsible for environmental protection. If liquidation or bankruptcy proceedings are opened against the holder of the exploitation, the costs of rehabilitation and recultivation of the land on which the exploitation was carried out will be covered as a matter of priority from the liquidation or bankruptcy estate.

Proposals for improved legal regulations in Serbia:

- To improve or specify certain norms, to formulate a proposal for amendments to the law, consolidate all measures for the implementation of reclamation into the law on mining as in German practice
- Policies and strategy development (emphasizes that coal mine closure requires clear policy direction, large budget outlays, and significant stakeholder consultations)
- Public participation is crucial for an effective system of environmental protection; however, the Environmental Impact Assessment Law does not provide details about what form public participation should take. It is necessary issue Interim Measures for Public Participation in Environmental Impact
- Tightening of the penal policy for non-implementation of reclamation solutions within the prescribed legal time
- Harmonization according to the EU legal framework.

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6 CONCLUSION

The changes in the environment effected by the coal mining are so complex that often no natural recovery of disturbed areas can occur. Land recultivation is a method of countering the negative after-effects of extracting coal and and returning the land to its original purpose or conversion land. It is also a comprehensive interdisciplinary process that covers the problems of landscape redevelopment, the restoration of its productivity, environmental quality, economic and aesthetic values.

The area of land reclamation damaged by surface coal exploitation is widely covered by legal regulations and that it is the responsibility of several ministries, more precisely their authorities. In practice, this creates a number of problems, especially in the phase of creating and approving project and technical documentation, because it requires more time and higher costs. Consequently, the field of reclamation should be regulated uniformly by the Mining Law, which would avoid unnecessary administration and would not affect the quality of technical solutions and their implementation.

Identifying development of the legal framework governing the restoration of disturbed land areas, the revealed stage-by-stage evolution of the legislative base of recultivation indicates a 10–year quality gap between the Serbian legislative framework governing recultivation and the foreign ones.

Based on all of the above, it can be concluded after mining exploitation and reclamation, land should also be regulated by national laws, effectively strengthen the protection of tillable land, promote the economic cycle of land, and realize the environmental value of mining land.

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UNDERGROUND MINING ENGINEERING 42 (2023) 37-45 UNIVERSITY OF BELGRADE - FACULTY OF MINING AND GEOLOGY UDK 62 ISSN 03542904

Original scientific paper

X-RAY STRUCTURAL ANALYSIS OF THE BaO AND TiO₂ STARTING COMPOUNDS AND INITIAL MECHANOCHEMICAL ACTIVATION

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Received: March 26, 2023

Accepted: May 08, 2023

Abstract: The aim of this research was to obtain an initial characterization of the BaO and TiO₂ components, as well as to conduct preliminary research into the possibility of activating the initial components in order to obtain barium titanate. Chemical and X-ray structural analysis of the initial components was performed. In order to determine the possibility of applying a mechanochemical activator and the activation time in which the first intermediate compounds were formed by the activation of the starting substances, the system was activated for 30 minutes. After this, an X-ray structural analysis was performed and certain changes in the crystal lattice of the starting components were made. X-ray structural analysis showed that during grinding, amorphization of the initial components occurred, which is the first stage in the process of obtaining titanate, as well as the presence of a small amount of barium titanate, which is indicating the presence of an intermediate compound in the process of finally obtaining barium titanate.

Keywords: mechanochemical activation, barium titanate

1 INTRODUCTION

Synthesis reactions that occur during mechanochemical activation are chemical reactions in the solid phase. The study of reactions in the solid state implies the existence of specific parameters characteristic of the complex state of solid matter (Korobov, 2005; Hokkaido University, 2019; Li et al., 2017; Naseer, 2005). In this type of chemical reaction, there is a direct contact of solid particles, without their dissolution and decomposition into ions. Reactions in the solid phase are only possible if they are

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thermodynamically feasible. A necessary condition is the initiation of the reaction, i.e. the supply of the required amount of energy. The initial energy must be the same or higher than the required activation energy for the desired reaction expected between the components and the conditions in which it takes place (Oleynikov et al, 1974; Gomes, 1961; DeBoer & Selwood 1954; Behrens, 2008; Criado, 1981).

Mechanochemical synthesis is solid phase reaction, without prior dissociation of reactants in the classical sense and without the mediation of solvents. As such, it cannot be based on known theories about acid-base reactions (neutralization reactions). If, as a product of a mechanochemical reaction, a chemical compound is obtained which, according to its chemical composition and other physical and chemical characteristics, is a salt of an acid and a base, and the reaction was entered with an anhydride of an acid and an oxide base of a hydroxide, such a reaction must still be considered a neutralization reaction. The mechanism of the neutralization reaction in mechanochemical processes could be set in such a way that some kind of dissociation is still ahead of the reaction, as a result of the collision of elementary particles of two reactants, where the collision must have enough energy to cause dissociation. By grinding, i.e. mechanical activation, energy is supplied to the system, interatomic bonds in the molecule are excited, bonds inside crystal lattice are broken and new states are established. At the moment of collision, the main role in the further course of the reaction is assumed by electrons from excited and broken bonds. The outcome of the reaction is conditioned by the establishment of new, stable bonds that will be built by those electrons - carriers of the reaction. In the case of mechanochemical reactions of neutralization, perhaps only Lewis' definitions of acid-base reactions (Lewis, 1916; Avvakumov, 1986), i.e. neutralization reactions, can be partially applied, in the domain of acid as an acceptor and base as an electron donor. The amount of energy required to initiate a chemical reaction has the greatest influence on the kinetics of chemical processes in the solid phase (Boldyrev, 1993).

Barium titanate is a product that can be obtained from barium oxide and titanium dioxide in a mechanochemical reactor. For the examination of the processes that occur during the reactions between substances in the solid state, and the examination of the kinetics and mechanism as well as the transition phases of intermediate compounds during the process of mechanochemical synthesis, a significant emphasis is placed on the reactions between titanium dioxide and the corresponding oxides, including barium oxide (Lazarević, 2009).

2 EXPERIMENTAL PROCEDURE

The mechanochemical activation of two oxides (BaO and TiO₂) was performed in a highenergy vibration mill with torsion springs and annular working elements, manufacturer KHD Humboldt Wedag A.G., Germany, type MH954/3. The grinding time was 30 minutes. The power of the activator motor was 0.8 kW. The mill consists of a bed and a horizontally placed shutter. The working vessel is a cylindrical vessel made of stainless steel, 40 mm deep and 170 mm inside diameter.

The working elements consist of two free concentric rings made of stainless steel with a total mass of 3 kg. The optimal amount of mechanochemically activated powders is 50-150 g. The samples were recorded and diffractometrically analysed on an automated diffractometer with a PHILIPS PW-1700 copper tube, which operates at 40 kV and 35 mA. The device is equipped with a graphite monochromator and a proportional counter, filled with xenon. The shooting angle (20) from 4 to 15° was used. For the experiments, barium oxide (BaO) was used as a basic reactant, and titanium dioxide (TiO₂) as an acidic reactant. No preconditions have been set for the starting components in relation to the crystal structure and granulometric composition. The optimal amount of background components that are activated in the mechanochemical reactor is from 50 to 150g. Recalculated according to reaction 1, the amount of starting substances consisted of 0.5 mol (76.7 g) BaO and 0.5 mol (39.9 g) TiO₂, so the total amount of starting material was 116.6 g.

$$BaO_{(s)} + TiO_{2(s)} = BaTiO_{3(s)} (BaO \cdot TiO_2)$$
(Eq. 1)

Barium oxide manufactured by FLUKA (Switzerland), CAS No. was used in the experiments. [1304-28-5], *pro analysi*. The chemical characteristics of BaO are given in Table 1.

Content (acidimetric), min.%	98
Substances insoluble in HCl, max.%	0.005
Impurities, max.%	
Carbonates (as CO ₂)	0.5000
Chlorides (Cl)	0.0050
Sulfates (SO ₄)	0.0010
Total Nitrogen (N)	0.0020
Arsene (As)	0.0001
Calcium (Ca)	0.0200
Iron (Fe)	0.0050
Potassium (K)	0.0050
Sodium (Na)	0.2500
Lead (Pb)	0.0010
Loss on ignition, max.%	0.5

Table 1 Chemical characterization of barium oxide

In order to identify barium oxide and its crystal structure, X-ray structural analysis was performed, shown in Figure 1.



Characteristic diffraction maxima of barium oxide

Pick no.	d - value	Angle 20
1	3.2895	27.085
2	2.6771	33.445

Figure 1 X-ray structural analysis of barium oxide sample

By analysing the X-ray results showed at Figure 1, it was established that it is a substance that is largely amorphized. Diffraction maxima (d-values 3.2895 and 2.6771) lead to the conclusion that the analysed sample corresponds to (poorly crystallized) barium oxide (chemical composition BaO), according to the ASTM standard (No. 22-1056).

TiO₂ manufactured by MERCK (Germany), CAS No. was used in the experiments. [13463-67-7], quality *pro analysi*. Table 2 shows the chemical composition of the titanium oxide used.

X-ray structural analysis of the BaO and TiO₂ starting compounds...

The content, %	99.0 - 100.5	
Substances soluble in water, max.%	0.5	
Substances soluble in HCl, max.% Impurities, max.%	0.5	
Heavy metals (like Pb)	0.0020	
Acid-soluble antimony (Sb)	0.0002	
Sulfates (SO ₄)	0.0010	
Total nitrogen (N)	0.0020	
Acid-soluble barium (Ba)	0.0002	
Arsenic (As)	0.0001	
Iron (Fe)	0.0050	
Acid-soluble lead (Pb)	0.0010	
Zinc (Zn)	0.0005	
Loss on ignition at 800 °C, max.%	0.5	
Loss on drying at 105 °C, max.%	0.5	

Table 2 Chemical characterization of titanium dioxide

X-ray structural analysis of the starting titanium oxide and characteristic diffraction maxima are given in Figure 2.



Characteristic diffraction maxima of titanium dioxide

Pick br.	d - value	Angle 2 O
1	3.5133	25.330
2	2.3778	37.805
3	1.8926	48.035
4	1.6667	55.055
5	1.4806	62.700

Figure 2 X-ray structural analysis of titanium dioxide sample

During the experiments, a sample was taken from the reaction system and its X-ray structural analysis was performed. The activation of the starting components initially lasted 30 minutes, in order to determine whether this time is sufficient to start the chemical reaction process in the solid phase. For the analysis of the chemical composition of the system, depending on the activation time of the starting substances, the atomic absorption spectrophotometric method was used.

3 RESULTS AND DISCUSSION

X-ray structural analysis of the mixture of starting components was performed after 30 minutes of activation. In this way, both starting components and products of mechanochemical processes are identified and monitored. Figure 3 shows the X-ray structural analysis of the sample after 30 minutes of mechanochemical treatment.



Characteristic diffraction peaks (20 peaks registered)

Pick br.	Characteristic for substance	d – value	Angle 20
1	BaTiO ₃	3,9940	22,24
2	TiO ₂	3,4937	25,47
3	TiO ₂	3,3878	26,28
4	BaO	3,3099	26,91
5	BaTiO ₃	2,8273	31,62
6	BaO	2,6794	33,41
7	BaTiO ₃	2,3231	38,73
8	BaTiO ₃	1,9461	46,63
9	TiO ₂	1,8848	48,24
10	TiO ₂	1,6545	55,49

(*The remaining 10 registered peaks belong to the noise domain*) **Figure 3** X-ray structural analysis of the reaction system sample (BaO+TiO₂) after 30 minutes of mechanochemical treatment

The broadening of the diffraction lines and their insignificant intensity indicate that after 30 minutes there was a significant disruption of the crystal structure of the initial reactants, especially titanium dioxide, whose crystal structure was very clearly expressed before the start of the reaction (Figure 2). The analysis of the registered peaks and the corresponding diffraction angles confirmed the presence of initial reactants, but also the

beginnings of the formation of the product - barium titanate at some of the characteristic diffraction angles for this substance.

Based on the diffractogram, it can be concluded that in these 30 minutes of activation (grinding) the first phase of activation took place and it is the phase of progressive destruction of the crystal structure of the reactants. It was also determined the existence of a compound that, according to its chemical composition, contains all the components of barium titanate, but it is still an intermediate compound, which indicates the fact that during this grinding time, the beginning of a chemical reaction between the initial components took place. The presence of pure oxides as well as traces of barium titanate indicates that activation in the sense of a solid-state chemical reaction has begun during this initial activation time.

Observing the reaction, the mechanical energy that is imputed into the reaction system, in this phase is mainly spent on the destruction of the crystalline structure of the initial reactants and their significant amorphization. The mechanochemical activation of individual substances (as well as the combination of two or more substances) leads to the transformation of the released mechanical energy and its accumulation in the treated material in the form of accumulated deformations of the crystal lattice, an extremely developed and active specific surface of the material, whereby the material is brought to an elevated level of energy content. This results in an increased degree of potential energy, i.e. chemical reactivity of the material.

Considering that the reaction was started with pure and inactivated reactants, which as elementary particles (BaO and TiO₂ molecules) exist as reactants during the mechanochemical reaction, it can be concluded that the first phase of the reaction takes place in the first 30 minutes. It involves mutual collisions between the molecules of two reactants, resulting in the formation of an activated complex, which stoichiometrically corresponds to the chemical composition of barium titanate, but in terms of structure and characteristics of chemical bonds, it represents a special compound. The second stage should be the further introduction of energy into the reaction system, which assumes that the energy barrier on the reaction path to the product of the reaction should be overcome, so the chemical reaction irreversibly flows towards the expected product.

In further experiments, it is planned to extend the grinding time, and to monitor the activation products, which would determine the exact time required in the given device for the starting components to completely chemically react and give the desired product, barium titanate.

Acknowledgments

This work was financially supported by the Ministry of Science, Technological Development and Innovation of the Republic of Serbia (Grant Nos. 451-03-47/2023-01/200023 and 451-03-47/2023-01/200026

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UNDERGROUND MINING ENGINEERING 42 (2023) 47-64 UNIVERSITY OF BELGRADE - FACULTY OF MINING AND GEOLOGY UDK 62 ISSN 03542904

Review paper

ROCK EXCAVATION METHODS IN URBAN AREAS

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Received: June 05, 2023

Accepted: June 20, 2023

Abstract: The increased volume of construction works, which includes the construction of new residential and business buildings, hotels, garages, shopping centers, and similar, is increasingly common in urban areas. Such works are increasingly coming into contact with different types of problems during construction, as one of these problems we single out the appearance of solid rock mass during excavation at the construction of facilities or part of the building, which includes the underground premises of a building, it is necessary to excavate the material on site, however, due to its physical and mechanical characteristics, it is not always possible to excavate this material with conventional machinery. The composition of the material that is removed is sometimes in the form of solid rock, and thus its excavation is more complicated, so other methods must be used: controlled blasting, application of expansive mortars, etc. This paper presents some of the possibilities of removing solid rock material for the purpose of smooth construction of buildings.

Keywords: urban areas, excavation, expansive mortars, controlled blasting, construction

1 INTRODUCTION

The technology of excavating rock material can be performed by various methods that require precise execution and a high degree of safety during the execution of the works in order to perform them without unwanted consequences.

Some of the methods that can be applied in order to remove the rock mass are:

- application of explosives (blasting),
- application of expansive mortars,
- possibility of cutting rock material (chain saw machines, diamond wire saw machines),
- application of hydraulic breakers or rippers.

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The mentioned methods can be used and are very effective. However, many factors influence the choice, such as the scope of work and the amount of rock material that needs to be removed, the expected and available time for excavation, the characteristics of the rock material, etc. Generally, smaller amounts of rock material and material with cracks can be easily crushed with the already-mentioned machinery (hydraulic breakers or ripper). Currently, there is a lot of equipment available on the market in the form of heavy hammers (breakers) and rippers, which can be mounted on hydraulic excavators. When the rock is solid and without cracks, a very efficient way of removal can be done with cutter machines, which are normally used in mines of decorative stone. The application of expansive mortars has the advantage that there is no noise, vibration, or other negative effects, which is also a problem with work in an urban environment and needs to be reduced to an appropriate level. The disadvantages of expansive mortars are the high costs and profitability of works when it comes to a large amount of rock material. In the case of a large amount of material (solid rock), the effective method is the use of explosives, but attention should be paid to all negative effects which can appear during the blasting. This method is mostly used in combination with the other mentioned methods. In this paper, we will present each of the mentioned methods in detail and the technology of excavating solid rock mass in urban areas during the construction of buildings.

2 CONDITION ASSESSMENT IN THE CONSTRUCTION AREA

Before the excavation of rock material needs to record the condition of the surrounding area, regardless of the choice of method, especially if it involves the use of explosives. When excavating the rock mass, there are negative effects that can occur, namely:

- noise and vibration,
- the appearance of dust and gases,
- blasting vibration,
- flyrock.

Most often, in the immediate vicinity of such places, where the work is being carried out, there are a large number of residential and business buildings, churches, or some landmarks (cultural monuments) and similar, therefore it is necessary to inspect them and record the current condition (in the form of photographs, documents...). During work, it is necessary to use compliance with the prescribed measures of protection and safety at work. Figure 1. shows an example in urban area.

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Figure 1 An example of excavation of rock material in an urban area (Zakladani, 2023)

3 TECHNICAL DESCRIPTION AND WORK TECHNOLOGY

The work on the excavation requires attention and precision, the conditions are a challenge for everyone, and it is necessary to follow all prescribed parameters and calculated values. When choosing the appropriate method for excavating rock mass in urban areas, it is important to consider factors such as safety, noise, vibration, limited space, and environmental protection. These techniques are often applied with appropriate precautions and technical experts to ensure the efficiency and safety of work in urban areas.

3.1 Application of explosives (blasting)

Drilling of boreholes, as well as contour boreholes, is carried out with a drilling diameter of 27 mm to 51 mm, therefore the use of explosives is reduced to cartridge explosives of smaller diameter and quantities that must be calculated beforehand. In order to carry out blasting works as safely as possible, it is necessary to follow the prescribed quantities of explosives per delay time during such works. Currently, various models of well-known manufacturers of this type of equipment such as AtlasCopco, Sandvik, etc. can be found on the market. Figure 2 shows one drills that can be used for drilling with small-diameter boreholes.



Figure 2 AtlasCopco drilling machine for small diametar (Epiroc, 2023a)

The application of this type of drilling machine or similar allows the production of small diameter boreholes and thus the use of a smaller amount of explosives in order to reduce the impact of harmful effects of blasting in urban areas such as the impact of seismic effects. This type of drill can also be used for drilling auxiliary boreholes that are used, for example. for threading the diamond wire and later cutting the rock material. Small-diameter wells can be used for the use of expansive mortars if there is a need for their application.

At the location of the works, it is necessary to define in advance the zones of the maximum permitted amount of explosives per delay time in relation to the surrounding objects. During blasting, work safety measures should be observed, during blasting it needs to stop all work, stop traffic if there is any, and give a sound signal before the initiation of the blast field, covering the blast field is also necessary before initiation (with blasting mats, etc.). Each borehole that is filled with the prescribed amount of explosives must properly do the stemming of the borehole in order for the blasting effect to be complete. Figure 3 shows different cartridge diameters of "Maxam" explosives.



Figure 3 Different cartridge diameters of "Maxam" explosives (Maxam, 2018)

The initiation of charging in the boreholes is done by the Nonel initiation system. The Nonel system provides adequate safety during initiation, reduction of seismic effects of blasting, a combination of different delay times, use in boreholes filled with water, etc. Initiation of Nonel detonators outside of boreholes can be done with an electric detonator, primer, or machines specialized for initiation of Nonel detonators (Savić, 2000). The detonating cord can be used when charging in the boreholes only inside the borehole, or if there is a danger that there will be a bad transfer of detonation between explosive cartridges, as well as when separating the charge inside the borehole, also it can be used for contour boreholes (Purtić, 1991). Figure 4 shows non-electric initiation system "Nonel".



Figure 4 Non-electric initiation system "Nonel" (Trayal, 2023)

Before initiating a blast field, it is necessary to cover the blast field with some protective mats in the form of blasting mats, etc. By covering the blast field, the possibility of flyrock during blasting is prevented (Figure 5).



Figure 5 Covering the blast field with protective mats (Quarry, 2023)

After blasting, it is necessary to load and transport the material to the predicted location and clean the work site for the preparation of the next blast field. The work on preparing the terrain for the drilling of blast fields needs to be coordinated with the construction work so that the dynamic of all works can be performed without problems. It is a very complex type of blasting and excavation of rock material, so all work must be done with attention. The size of the blast fields, that is, the amount of blasted material must also be in accordance with the calculations of the blasting parameters, and the duration of the blast series must not be longer than 1s. The quantities of explosives per delay time must be in accordance with the project to obtain the best blasting results and reduce the negative effects on the environment.

3.2 Application of expansive mortats

Expansive mortars were created from the construction industry's need to destroy various concrete structures in urban areas without explosives and explosions. Very quickly they found application in mining when splitting and separating primary blocks in decorative stone mines. There are a large number of manufacturers of expansive mortars on the market, but mostly they are chemical agents with similar characteristics (Maksimović, 2006).

N^0	Ambient temperature (°C)	The speed of reaction, slow (h)	The speed of reaction, fast (h)
1	50	8	3
2	40	12	4
3	30	15	5
4	20	20	10
5	10	30	15

 Table 1 The speed of reaction for expansive mortars depends on the temperature (Maksimović, 2006)

First of all, by expanding in the borehole, they develop a pressure force in the range of 7-8 (kN/cm^2), which is much more than the necessary force for splitting the rock material. The speed of expansion directly depends on the temperature of the rock material, that is, the external air temperature, and it is proportional to the temperature. Fast reaction mixtures are 20% - 30% less powerful and this should be taken into account when determining the distance between the boreholes (Maksimović, 2006).

Expanding mortars have the following advantages:

- the rock mass splitting process is "quiet" without vibrations and explosions,
- by initiating the chemical reaction, or the process of spreading the mortars, the need for the presence of workers is not necessary,
- absolutely clean ecological product and very acceptable from the ecological aspect,
- safe storage,
- the use and preparation of the mortars are very simple and safe for humans, normally with the use of safety gloves and protective glasses (Maksimović, 2006).

The disadvantages are as follows:

- it is more expensive than classic explosives, but the price is slowly approaching the price of classic explosives,
- has limited application in cold and continental climates, because it cannot be used at temperatures below -5 °C,
- application in a tectonically cracked material is limited, it requires a solid rock material (Maksimović, 2006).

As for the method of use, the expansive mortars are mixed with water in an approximate ratio of 3:1. When the mixture is obtained, without blobs, it can be put into the borehole. Considering that the mortars are of alkaline composition, it is necessary to perform all operations with safety gloves and possibly use glasses. Boreholes are protected from contact with water, or rain, by covering them with PVC film (Maksimović, 2006).

Table 2 The required amount of expansion mortars according to the borehole diameter (Maksimović, M., 2006)

1	Borehole diameter (mm)	30	40	50
2	Amount of powder (kg)	1,1	2,0	3,1
3	Amount of mortars (kg)	1,4	2,6	4,0

Expansive mortars have the following advantages over other technologies for the destruction of rock material:

- completely noiseless rock splitting,
- do not generate smoke, dust, and gas,
- no vibrations and negative effects,
- do not cause damage to the rock mass (Maksimović, 2006).

3.2.1 Application of expansive mortars for making protective screen

Blasting works must be carried out mainly with the prior preparation of a free surface around the work site in the direction of the buildings located near it. That free surface represents a type of protective screen that will reduce the impact of blasting vibration on the environment (Kričak, 2006). The rock mass will therefore be interrupted by a crack, artificially created in order to reduce the resulting waves to the appropriate limit during blasting within the work site. This would reduce the problem of blasting vibration after blasting, as well as the impact of blasting in order to remove rock material from under the horizontal concrete pav and around the pillars. Figure 6 shows an example of making vertical contour holes to create a protective screen.



Figure 6 An example of making vertical contour holes to create a protective screen

During construction works, a lot of supporting pillars or piles are installed on the construction site, so blasting around them is risky. It is also necessary to make some kind of crack around the columns with contour boreholes in order to reduce the impact of blasting vibration on the pillars, as shown on figure 7.



Figure 7 Example of creating contour boreholes around pillars

By creating a protective screen, or free areas around the work site or in locations where the work takes place under the concrete pav, around pillars and piles, and the drilling of boreholes on the blast field, through which we will gradually remove certain amounts of rock material, can be started. The diameters of boreholes that need to be drilled are generally smaller diameters, as well as the use of smaller diameter explosives, due to the limitation of initiating the amount of explosive charge per delay time.

3.3 Possibility of cutting rock material (chain saw machines, diamond wire saw machines)

Cutting machines are good for extracting smaller blocks from the rock mass or forming an ideally flat wall in the rock mass of the foundation pit. They can also be used when creating a protective screen that will reduce negative effects (blasting vibration) on surrounding objects in case of rock material blasting. They are not suitable for large amounts of rock material because the excavation would take a long time due to the very technology of these machines. As a result of the work, stone blocks are obtained, which need to be subsequently crushed and then loaded, which further complicates the process. A diamond wire saw machine is better for the application as it covers larger cutting areas in one go as well as multiple cuts from one spot. Chain saw machines require a flat surface for movement and frequent movement which further slows excavation.

3.3.1 Chain saw machine

Chain saw machines (Figure 8) are used to obtain blocks of decorative stone, but also cut stone. The machine consists of a base (1) on which a motor block (2) is installed, connected to a chain (4). The chain (3) moves along the groove of the chain. The base moves on rails (5). The engine block consists of three hydraulic systems. One is used for driving (turning) the arm (console) of the cutter, and the third - is for moving the base (undercarriage) along the rails. Each hydraulic system can work autonomously, or with a single engine (Popović, 2013).



Figure 8 Chain saw machine (Popović, 2013)

The hydraulic system of the machine enables continuous regulation of the cutting speed, pressure, and cutting of the chain, the result of which is the optimization of the working mode of the machine on rock materials with different physical and mechanical properties. For making horizontal (underground) cuts, the cutting assembly is fixed on the additional support, which enables the cut to be made at the floor level (trimming) (Popović, 2013). Chain saw machine developed by FANTINI is shown on figure 9.



Figure 9 Chain saw machine by Italian company FANTINI (Fantini, 2023)

3.3.2 Technology of chain saw machine operation

Cutting with a chain saw machine, the maximum cutting depth is about 2m. A horizontal cut with a chain saw machine is simpler, because the cutting waste flows away with the water, while it is difficult with a vertical cut, so a large part of the chain's energy is spent on removing the cutting waste, instead of cutting. Therefore, compared to the chain saw machine, the diamond wire saw machine has some advantages. In surface mines of decorative stone during excavation, these two machines usually work in combination (Popović, 2013). The technology of making a vertical cut with a chain saw machine machine without making a cut beforehand is shown on figure 10.



Figure 10 The technology of making a vertical cut with a chain saw machine without making a cut beforehand: a- chain cutting; b- working stroke of the machine in a vertical cut; c- completion of the cut; d- turning the chain (clockwise) and new chain cutting; e- working stroke of the machine in the anticlockwise direction and finishing the vertical cut (Popović, 2013)

3.3.3 Diamond wire saw machine

Cutting with a diamond wire saw machine is a procedure that has become established and has almost become dominant in relation to other machines in the exploitation of decorative stone. When it comes to igneous rocks, there are objectively more factors that make cutters with diamond rope more suitable than other machines (Maksimović, 2006). The advantage of using this machine for the purpose of making a protective screen is that a larger cutting surface can be covered with a rope than with a chain saw machine.

Good performance, good impacts on the working and living environment, flexibility in handling, and a wide range of applicability of the machine, make the diamond wire saw machine best suited to the requirements of a wide range of destruction of rock materials and trends in the protection of the environment. The destruction of the rock material takes place in a closed-cutting system and can be realized in the following ways:

- the convergence of two boreholes,
- by cutting a borehole with one cut plane,
- by the intersection of two cut planes (Maksimović, 2006).

By mechanical structure, most cutting machines consist of the following basic parts:

- undercarriage frame,
- motor part,
- dashboards (Maksimović, 2006).

The undercarriage is a steel construction of the machine that enables safe, secure, and long-lasting operation of the machine. The undercarriage is structurally solved in different ways depending on the manufacturer. Basically, all constructions carry the motor part of the machine with the drive wheel, as the most important part of the machine (Maksimović, 2006).

The cutting mode can be adapted to the physical-mechanical characteristics of all rock materials. The speed regulation is especially important when starting and ending a cut. A good cutting regime can achieve an optimal relationship between the performance and durability of wire (Maksimović, 2006).

The most important features of a diamond wire saw machine are:

- technical performance of the machine,
- construction and type of steel wire rope with diamond wire rope (Maksimović, 2006).

During operation, the machine moves on a frame or rail track, which can have different profiles (square, triangular, and tubular). The frame consists of rails or rail segments of different lengths (usually 3 m). It contains various components and moving devices and serves to support all the connecting installations (such as a derrick with guides, and a guard for the steel rope, which can be unwound for protection during cutting). The statics, as well as the dimensions of the rail track, are different in different models, which is logical because they carry motor construction of different powers (Maksimović, 2006). Diamond wire saw machine is shown on Figure 11.

The exceptional operability of a diamond wire saw machine is achieved by a set of auxiliary devices, guides, and rollers that technologically create the conditions for changing the direction of cutting, achieving a certain geometry, and any technological requirement when it comes to cutting (Maksimović, 2006).



Figure 11 Diamond wire saw machine by Italian company LOCHTMANS (Lochtmans, 2023)

3.3.4 Technology of diamond wire saw machine operation

There are several ways to perform the primary cut. In specific conditions, a technological solution that corresponds to the specific situation will be adopted. In principle, the following methods of performing cuts are applied:

- 1. horizontal cutting with a "loop",
- 2. vertical cutting with a downward "loop",
- 3. vertical cutting of back cuts with a guide,
- 4. vertical cutting with an upward "loop",
- 5. vertical cutting of the "blind" cut (Maksimović, 2006).

Working diagram of diamond wire saw machine is shown on figure 12.



Figure 12 Working diagram of a diamond wire saw machine (Maksimović, 2006)

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3.4 Application of hydraulic excavator with breakers or rippers

Fragmentation of the material can be performed by a mechanical process, or with an impact hammer mounted on a hydraulic excavator. An excavator with an installed hydraulic hammer is a machine that is mainly used for auxiliary work on surface mines, such as crushing larger (oversized) pieces of rock. Fragmentation of material with this type of machine or a hydraulic excavator with a ripper is mainly used for auxiliary fragmentation and unapproachable parts related to work around supporting pillars, and under supporting structures where blasting cannot be applied. Of course, these machines require a lot of time to be able to crush compact rock, so they are mainly used for smaller amounts of material in inaccessible locations, auxiliary work, and crushing larger pieces of rock. Figure 13. shows the operation of a hydraulic excavator with a hydraulic hammer.



Figure 13 Operation of a hydraulic excavator with a hydraulic hammer (Epiroc, 2023b)

A hydraulic excavator with a ripper (Figure 14) is a machine that is intended for breaking rock material, it is similar to a hydraulic excavator with a hammer, with the difference being that the ripper produces shocks due to vibrations generated in the mechanism itself. The ripper is made of wear-resistant steel, it makes less noise than other machines designed for the same purpose.



Figure 14 Hydraulic excavator with the ripper (Xcentric, 2023)

During work on the removal of the rock mass after its fragmentation by one of the methods, it is necessary to load and transport the material outside the work site to the designated place. For this purpose, trucks are used for transport, and hydraulic excavators are used for loading at smaller locations and in urban environments. In accordance with the scope of work and the quantities that need to be calculated, the required number of trucks and loading machines must be calculated, these works must be in accordance with the dynamics of rock material excavation at the site.

4 CONCLUSION

The excavation of rock material is a complex excavation procedure in an urban environment. The works are taking place in the nearness of existing buildings and safety and health measures at work must be observed in order to reduce negative impacts on people and the environment, as well as the safety of workers at the work site itself.

When there is a large amount of material that needs to remove in order to be able to continue with the construction work on the site, the effective excavation method for the rock mass would be a combination of the mentioned methods, for example, the application of controlled blasting with the use of expansive mortars, where for this purpose to have some drilled boreholes either for explosive or mortars. Also, the application of different types of cutting machines also requires the production of smaller diameter boreholes, for example, in order to pass a diamond rope if that method is used. The above-mentioned mechanization can be used as a secondary method and according to some calculations, the best efficiency would be obtained.

When carrying out blasting, it is necessary to ensure all conditions for the safe initiation of the blast field, such as notification of blasting, stopping of traffic, sound notification before the initiation of the blast field, and others. The method of blasting rock material with explosive means is perhaps the cheapest form of rock mass excavation. However, this method must be combined with some of the presented methods in order to create screens that would protect the surrounding objects, because most of the work sites are in urban areas.

Through this paper, some of the possibilities of excavation rock material in the urban environment are given, where a combination of these methods can be used to obtain an efficient way to carry out the work.

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