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Research paper

Study of the Influence of Sensitizer Content on the Density of a Bulk Emulsion Explosive Used in Underground Operations

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Abstract: Emulsion matrix sensitization is typically performed by chemical reduction of the density using different sensitization agents. Mixing of the components takes place directly inside the loading hose, which is equipped with static mixing devices. Precise dosing of the components, due to the multi-ingredient nature of the mixture, has a significant impact on the detonation and operational parameters of the end product. However, the operation and maintenance of the mixing-charging units in underground mines is very difficult due to the local conditions. As a consequence, different values for the detonation parameters may be expected when charging the same explosive into blastholes using two different charging units. The present article presents the results of laboratory testing of the influence of the sensitizing agent content on the density of a bulk emulsion explosive. Analysis confirmed that increasing the concentration of the sensitizing agent by 30% and 50% led to density reductions by 4% and 7%, respectively. In turn, reducing the sensitizer content by the same percentages resulted in an increase in the final density by 7% and 8%, respectively.

Keywords: blasting, emulsion explosives, density measurements, chemical sensitization

1 Introduction

1.1 Emulsion explosives

Already in 1964, intensive research on slurry explosives had led to the patenting of the first composition of ammonium nitrate (AN)-containing emulsion sensitizers by the American chemists Richard Egly and Albert Neckar [1]. The developed water-in-oil emulsion explosive consisted primarily of an aqueous AN solution and fuels, plus additions like AN prills that made the emulsion capable of detonation. Later in 1969, Harold Frederick Bluhm [2], from Atlas Chemical Industries, patented the water-in-oil emulsion explosive, in what is commonly accepted as the date for the discovery of emulsion explosives. Currently, this type of explosive is primarily used worldwide for hard rock extraction in both underground and surface mining, but also in civil engineering, tunnelling and demolition. According to the forecast by Future Market Insights [3], the global sales of emulsion explosives is projected to reach 13 million tonnes by the end of 2029.

In most cases, attempts at mechanizing the process of solid rock extraction have not reached a successful level thus far [4]. In fact, certain solutions exist, but their implementation on an industrial scale is not economically feasible [5]. It can therefore be predicted that the use of explosives, including emulsion explosives, will remain at a comparable level in the coming years. A similar trend can be observed in Polish underground copper mines, where the current annual consumption of AN-based emulsion explosives varies between 16 and 18 thousand tonnes, of which over 70% are bulk emulsion explosives. The blastholes in these mines are loaded using mixing-charging units installed on blasting utility vehicles. Explosives are produced directly at the point of loading using these mixing-charging units. These units mix the emulsion with additives (gassing additives) which produce small bubbles in the final products (and the density changes due to the increase in volume). These small bubbles, which are known as “hot spots”, are able to change the detonation properties of the emulsion. Further details on the physics of this hot spot process can be found elsewhere [6]. The mixing of the emulsion and the gassing additives takes place inside the loading hose, which is equipped with static mixing devices in the form of cross-stream static mixers. Unfortunately, consistent delivery of the explosives by the mixing-charging units and their operators is very difficult due to the conditions present underground, of which the most important is a high rock mass temperature (up to 55 °C locally), high air temperature (above 30 °C) and extremely high humidity (above 95%). As a consequence, completely different results for the velocity of detonation (VOD) and fragmentation may be observed

when loading the same explosive into blastholes using two different units. This problem is also associated with the operation of piston pumps, mainly when the blasting vehicle is travelling between the faces and panels (stuck and air locked pistons). In such cases, they do not dose the sensitizer properly. This results in a discontinuity of the lubricating film, which increases the pressure in the loading hose. Finally, smaller amounts of sensitizer result in a higher final density.

The density of an emulsion explosive has a direct impact on its sensitivity and explosion capability, as well as on the efficiency of blasting operations [7-9]. Due to the scale of the blasting operations in Polish copper mines, explosives are detonated at almost 700 faces daily. This raises doubts as to whether all of the blastholes are fired at densities that are optimal for a given explosive. Operational matters are also important, since the explosives may be fired from 30 min up to 48 h after loading. Such a long period of time between the loading and firing of the explosives has a very negative influence on their detonation performance [10, 11].

The idea for the investigations described herein arose from a study of the influence of time on the density of a bulk emulsion explosive performed in 2018 in an underground Polish operation. These tests confirmed a significant relationship between these parameters [12]. In the framework of the research, 40 samples of bulk emulsion were collected from 4 randomly selected mobile mixing-charging units (10 samples from each unit). The tests were based on density measurements at selected time intervals after loading. Analysis demonstrated that the initial density values varied for samples collected from each unit. Moreover, the density of samples collected from each unit decreased at a different rate. It was also found that the final density was reached at different times. In one case, no change in the density over time was observed for any of the samples from one of the mobile mixing-charging units, which indicates incorrect mixing of the emulsion with the gassing additives (also known as the “sensitisation process”). Therefore, the present authors have attempted to assess the influence of the sensitizer content on the density of a bulk emulsion explosive under laboratory conditions.

1.2 Sensitization of emulsion explosives

Emulsion explosives consist primarily of oxidisers, water, fuels, emulsifiers as well as sensitizers and modifiers of their physicochemical properties [13]. The emulsion matrix itself, consisting of an oxidiser and fuel phase, is not capable of detonation, and therefore needs to be sensitized. This can be achieved by adding plastic microballoons or glass microspheres (physical sensitization) or the appropriate chemical compounds (chemical sensitization). Chemical

sensitization can be performed in many ways by means of chemical reactions that generate gaseous products evenly distributed in the emulsion [14]. The most popular method is based on the reaction of sodium nitrite with AN (present in the emulsion). After mixing, the sodium nitrite and the matrix, the following reaction occurs:



The ammonium nitrite formed is very unstable and decomposes in an acidic environment, giving off nitrogen, as follows:



Since the saturated AN solution is trapped in the organic continuous phase and its contact with the sodium nitrite is hindered, the diffusion process is very slow, and thus a high component temperature is required to ensure proper kinetics of the system. To accelerate the reaction at lower temperatures, the addition of a nucleophilic activator, in the form of thiourea, should be applied:



This also leads to further reactions by the diffusion of the reagents, as well as their intermediate forms, through a thin oil film [6]. Furthermore, the nitrous acid molecule undergoes decomposition with the release of gas:



Based on the above formulas, one may conclude that reaction in the emulsion sensitization is a multi-stage process, and that each stage involving emission of gaseous products affects the final density. The exact course of these reactions is not fully known, and their order and rate are influenced by numerous factors, such as the type and content of the emulsifier (the thickness of the oil film determining the speed of the diffusion process), the degree of dispersion (characteristics of the production plant), the acidification method, the influence of salt additives in the oxidizing phase, the type and content of the buffering agent, *etc.* Overall, it can be stated that the process of sensitization using sodium nitrite for various emulsion matrix formulations is complex and

strictly unique, depending on the ingredients used and the technical parameters of the manufacturing plant.

In some cases, the abovementioned optimisation measures on the rate of the sensitization process are insufficient. In open-pit mining, where the rock mass temperature varies widely with the season, an additional acidifier, such as an acetic acid solution during winter, should be utilised in order to accelerate the sensitization reaction. Then, the reaction takes place outside the emulsion structure, avoiding the diffusion processes, according to the following formulas:



The excess of nitrogen oxide is released outside the reaction zone, where it forms nitrogen dioxide by reaction with oxygen from the air, visible as brown smoke in the area of the blasthole, as per reaction R9:



In turn, additional acidification is avoided in underground mining in order to decrease the toxic products (nitrogen oxides (NO_x) released from the gassing process). A single sensitizing agent is used, which is mixed into the emulsion matrix using the mixing-charging unit. This reaction occurs in the entire volume of the mass, and results in uniform gassing of the mixture and activates the matrix [15]. The most important operational parameter in the case of chemical sensitization is the rate of gas bubble production, and thus the time required to achieve the required final density of the emulsion.

In practice, the kinetics of this reaction depend primarily on the pH of the reagents, temperature and concentration of the active components. Industrially, the pH is already regulated at the preparation of the oxidiser solutions stage. The safety constraints for handling large amounts of acidified AN allow for a slight and strictly controlled reduction of the pH, as there have been cases of self-decomposition of hot acidified AN solutions in the past [16].

The temperature of the reaction in mines depends primarily on the rock mass temperature, as the reaction is initiated after mixing of the components and loading into the blasthole. The change in rock mass temperature affects the speed of sensitization. Moreover, in many cases the firing of the explosives is delayed due to unpredictable situations related to the complex nature of mining operations.

During that time, the density of the emulsion explosive forming in the blasthole is changing, and leads to changes in VOD.

The concentrations of the reagents are strictly defined according to the formula specified by the components manufacturer. The proportions of the components may change during working due to the difficult operating conditions of the dosing systems. The proportions, and thus the concentrations of the reagents, affect the speed of the sensitization reaction, but their influence on the final density is much greater. This is also a problem from a technical point of view, as the flow of the sensitizer in the loading hose serves as a lubricant coating of the inner surface of the hose in order to improve the flow of the matrix. Feeding of insufficient amounts of the sensitizing agent may result in clogging of the hose with the emulsion and blocking of the entire system. In such a case calibration of the unit is required in order to verify whether the desired volumes of matrix and sensitizer are dispensed at specific pump rotations.

Keeping the mixing-charging units in good working order is very difficult due to the conditions present in the mine described earlier. This is particularly true for underground mining. It may therefore lead to situations where the components of the emulsion explosive are pumped into the blastholes in the wrong proportions. This has a direct impact on the sensitization and the VOD (and potentially the detonation pressure) of the end product [17].

2 Materials

The first part of the study was to verify the density values of bulk emulsion explosives measured underground by shotfirers. According to the implemented procedure, each loading of blastholes must be preceded by a sensitization test. The density is determined by dividing the weight of the sample by the volume of the cup. During this test, a plastic cup is filled with the mixture of the matrix and sensitizer. While gassing, the emulsion is levelled with the top edge of the cup and weighed. The result is the value of the density 30 min after loading, which is noted in the form of a face charging report. The result is acceptable if the density value lies within the defined range, according to the applicable instruction.

The present analysis covered results of the density measurements obtained over a period of 4-5 months and included 4 randomly selected underground mixing-charging units. The number of measurements for each unit was different, as it was dependent on the frequency of the tests. Thus, the analysis included:

- 219 tests for unit #1,
- 184 for unit #2,

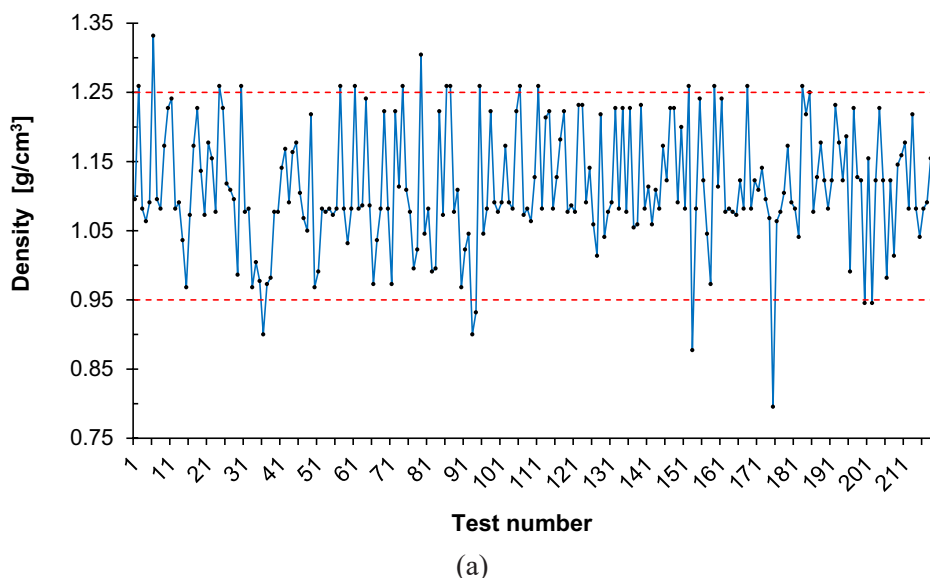
- 212 for unit #3, and
- 232 tests for unit #4.

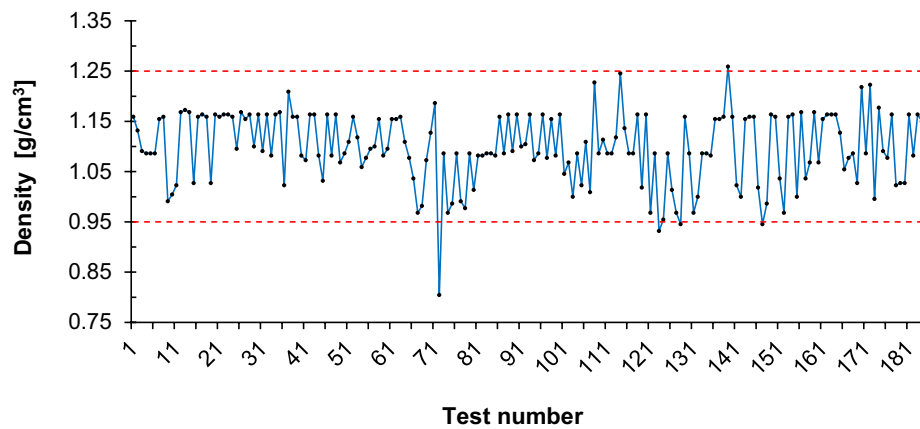
The results are shown in Figure 1, where the horizontal axis represents the successive tests. The red dashed horizontal lines are the minimum and maximum densities for the considered explosive, which should be within the range of 0.95 to 1.25 g/cm³.

Analysis confirmed a significant dispersion of the emulsion densities, both when comparing the individual units, and in terms of the values obtained for each unit during the period considered. In total, nearly 4% of the results were outside the required range. Particularly unfavourable results were observed for unit #1, for which as many as 22 values were outside the correct density range, and means a deviation of 10%. The average densities were as follows:

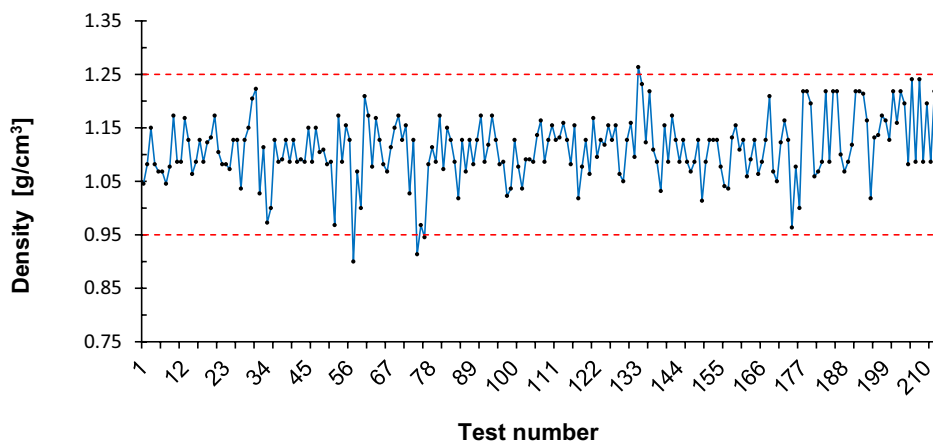
- 1.11 g/cm³ for unit #1,
- 1.10 g/cm³ for unit #2,
- 1.11 g/cm³ for unit #3, and
- 1.14 g/cm³ for unit #4,

which gives an average density of 1.12 g/cm³ (for all tests and units). The standard deviation of the results ranged from 0.06 g/cm³ for unit #3 to 0.09 g/cm³ for units #1 and #4 (0.08 g/cm³ for the overall data).





(b)



(c)

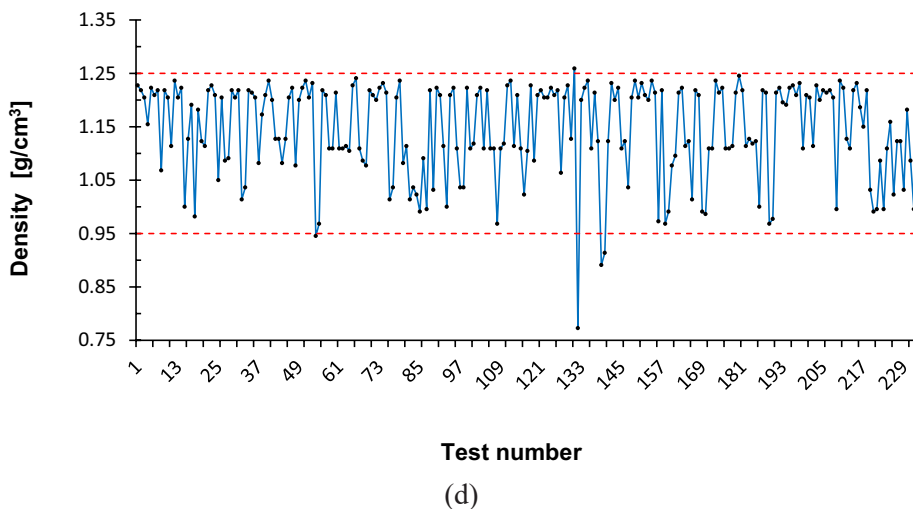


Figure 1. Results of density measurements for the selected units #1 (a), #2 (b), #3 (c) and #4 (d)

This indicates that sensitization under underground conditions is not fully controlled. Consequently, different densities may be observed when loading the same explosive into a blasthole using different units. This means that completely different VODs may be expected. Moreover, this may have a significant impact on the rock fragmentation. This can result from incorrect blending of the components in the loading hose. Inappropriate component dosing may also be an issue, since it can lead to the acceleration or deceleration of the sensitization. In such cases, calibration of the mixing unit is required.

It should also be highlighted that an incorrect density of an emulsion explosive may affect propagation of the detonation wave and cause other issues, such as partial burn or misfire, as such an explosive does not contain the necessary amount of reacting hot spots. Low-density emulsions are characterised by a high initiation sensitivity. Such explosives are less energetic than high-density ones, which in turn are characterised by lower sensitivity but higher detonation velocity and concentration of energy [18].

Based on the above, the authors have attempted to pursue the study under laboratory conditions, the purpose of which was to determine the influence of sensitizer content on the density of a bulk emulsion explosive. Laboratory testing has allowed other factors present at the firing site to be eliminated. It was assumed that this type of research would verify whether sensitization of an emulsion matrix, consisting in changes of density over time, is a fully controllable process. The tests were based on the Emulinit bulk emulsion

explosive manufactured by NITROERG S.A. (Poland), the same one as used for the density tests from different units (presented earlier). Selected parameters of the tested explosive, according to the EU-type examination certificate, are shown in Table 1. All of these were obtained for densities in the range of 0.95-1.05 g/cm³. Unfortunately, the authors did not have access to the formulation of the sensitizer solution due to the manufacturer's proprietary information.

Table 1. Selected parameters of the tested explosive

Parameter	Value
Critical diameter [mm]	34
VOD [m/s]	3,800 ^{a)}
Oxygen balance [%]	0.05
Trauzl lead block test [cm ³]	225
Friction sensitivity [N]	360
Impact sensitivity [J]	>30
Energy [kJ/kg]	3,546 ^{b)}

^{a)} for a diameter of 40 mm (unconfined VOD measurement), ^{b)} for a density of 1 g/cm³

The explosive samples were prepared using matrixes differing in storage times following their collection from the production line, including matrix M-1 collected 21 days before testing, matrix M-2 collected 7 days prior to testing and matrix M-3 collected 1 day before testing. This allowed the influence of matrix storage time on sensitization to be assessed. The matrix temperature was stable at 24.5 °C. The samples were prepared in the plastic cups. The analysis covered a standard dose of sensitizer for the tested explosive, as well as doses reduced by 30% and 50%, and doses increased by 30% and 50%. Six samples were prepared for each of the three matrixes and each sensitizer dose, giving a total of 90 samples. The first step was to measure the required doses of matrix and sensitizer. For this purpose, an electronic laboratory balance and plastic cups were used (Figure 2). The components were blended manually in 500 mL plastic cups using a glass rod. The blending time of a single sample was 30 s. After that, the mixture was poured into a 115 mL plastic cup. The sensitization time was controlled independently for each sample. As the volume of the matrix mixed with the sensitizer increased as a result of the chemical reaction, the excess of emulsion was scrapped off from the top of the cup and the samples were weighed at 5-min intervals for 60 min using an electronic laboratory balance. The samples were additionally weighed after 24 h. A view of the samples during the sensitization is presented in Figure 3.

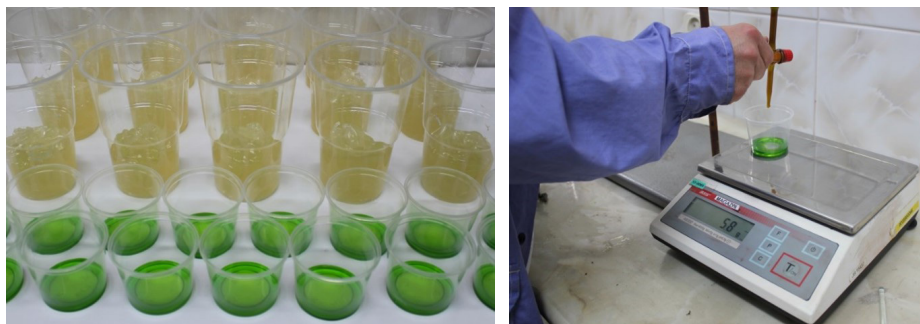


Figure 2. Preparation of the emulsion components



Figure 3. Selected samples during sensitization

The relevant matrix/sensitizer ratio for the Emulinit explosive was 95.5% to 4.5% (by mass). The modified sensitizer contents for the purposes of the tests were:

- 2.25% (–50% in relation to the standard content),
- 3.15% (–30%),
- 5.85% (+30%), and
- 6.75% (+50%).

3 Method

The explosive density (ρ) was determined based on the ratio of the net sample mass to the cup volume, according to the following formula:

$$\rho = \frac{m}{V} \left[\frac{\text{g}}{\text{cm}^3} \right] \quad (1)$$

where: m – sample mass (without cup) [g], V – cup volume [cm^3].

4 Test Results

Due to the large number of measurements and the repeatability of the results for individual samples within a given series, the analysis involved the average values for 6 samples (Table 2). The analysis showed that the density values for the samples prepared from matrixes collected from the production line at different time intervals are very similar. This indicates that the storage time of the matrix does not affect the sensitization. However, this only applies to the considered period of time, *i.e.* three weeks. An analysis of longer storage times of matrix is not justified, as the average consumption of bulk emulsion explosives in Polish copper mines is about 40 tonnes per day. Given such a high demand for explosives, the time between the production of the matrix and underground firing does not usually exceed 7 days.

The analysis confirmed the clear influence of the percentage content of the sensitizer on the density of the bulk emulsion explosive and the changes in density over time. Reducing the sensitizer content by 30% resulted in an average increase in the density of the end product by 6-8% (0.07 g/cm^3) after 60 min. Further reduction of the sensitizer content to 2.25% led to an increase in density to 1.10 g/cm^3 . In turn, increasing the percentage sensitizer content from 4.5% to 5.85% resulted in a decrease in density by 0.04 g/cm^3 . The clearest downward trend can be observed for the highest sensitizer content (+50%), which resulted in a decrease in density to $0.93\text{-}0.94 \text{ g/cm}^3$. In this case, sensitization is faster, which caused that emulsion to become oversensitized.

The results of these measurements are also presented in graphical form in Figures 4-6. In these cases, the density values were also averaged for samples from the individual measurement series. The results indicated that sensitization in each case progressed in a similar way. This confirmed that the manual blending of components had no negative impact on the course of the chemical reaction. The trend of the decrease in density over time was very similar for each matrix tested. The same applied to the trend in density decrease between different sensitizer contents.

Table 2. Average results of the density measurements

Matrix	Time [min]	Density [g/cm ³] for sensitizer content [%]				
		2.25	3.15	4.50	5.85	6.75
		(-50%)	(-30%)	Nominal value	(+30%)	(+50%)
M-1	5	1.31	1.33	1.31	1.29	1.28
	10	1.26	1.27	1.24	1.22	1.21
	15	1.23	1.23	1.19	1.17	1.16
	20	1.22	1.20	1.17	1.14	1.11
	25	1.20	1.19	1.14	1.11	1.09
	30	1.18	1.18	1.12	1.09	1.06
	35	1.17	1.17	1.10	1.07	1.04
	60	1.11	1.09	1.02	0.98	0.94
	1440	1.02	0.92	0.79	0.71	0.67
M-2	5	1.31	1.34	1.31	1.29	1.28
	10	1.27	1.28	1.25	1.22	1.22
	15	1.23	1.22	1.19	1.17	1.16
	20	1.22	1.21	1.16	1.13	1.12
	25	1.19	1.18	1.15	1.11	1.09
	30	1.18	1.17	1.12	1.10	1.07
	35	1.17	1.16	1.11	1.07	1.03
	60	1.10	1.09	1.01	0.97	0.93
	1440	1.01	0.91	0.80	0.70	0.65
M-3	5	1.31	1.33	1.30	1.29	1.29
	10	1.27	1.27	1.24	1.22	1.22
	15	1.23	1.23	1.19	1.17	1.16
	20	1.22	1.21	1.16	1.13	1.12
	25	1.20	1.19	1.14	1.12	1.10
	30	1.18	1.17	1.12	1.09	1.07
	35	1.17	1.16	1.10	1.06	1.04
	60	1.10	1.08	1.02	0.98	0.94
	1440	1.02	0.93	0.78	0.71	0.65

The explosive densities after 60 min ranged from 0.93 g/cm³ for the increased content of sensitizer to as much as 1.11 g/cm³ for the lowest content. Particularly important are the values of density obtained after 60 min from the beginning of sensitization and with increased content of the sensitizer. This indicated that the density was within the lower range of the value defined as correct and may suggest that the explosive will be characterised by a lower VOD.

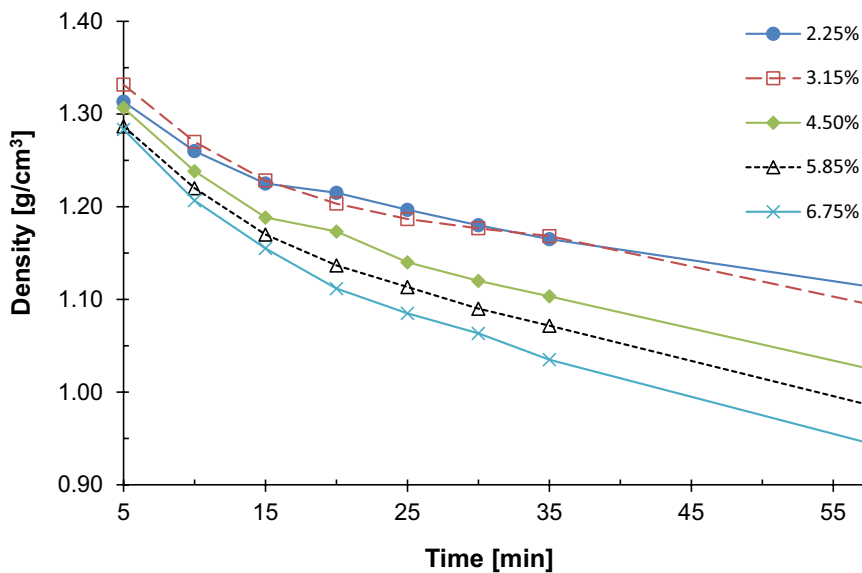


Figure 4. Changes in density over time during sensitization (matrix M-1)

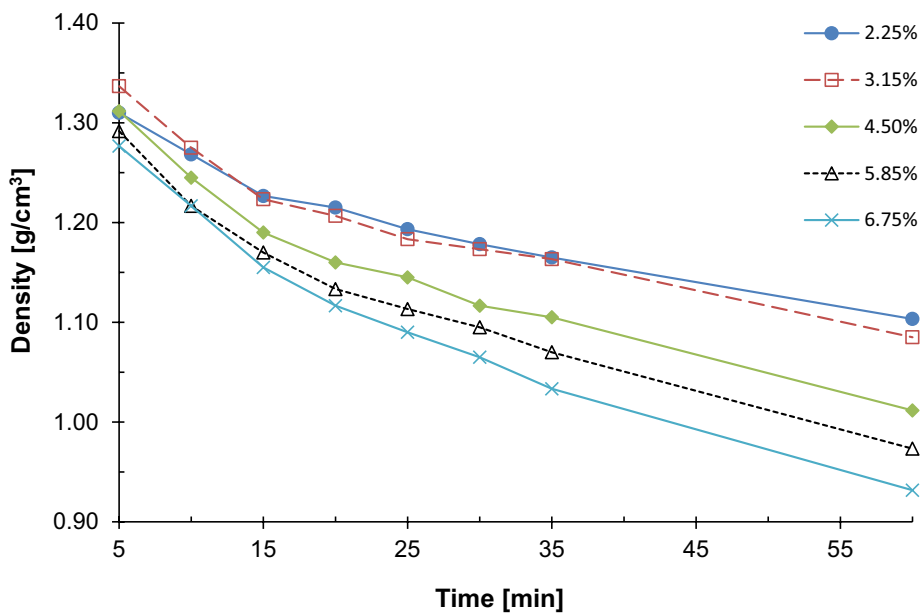


Figure 5. Changes in density over time during sensitization (matrix M-2)

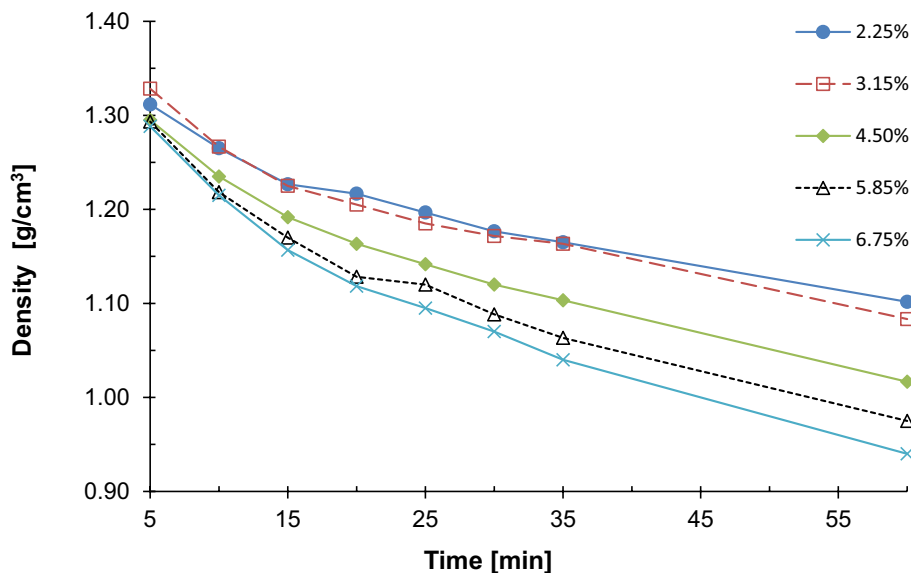


Figure 6. Changes in density over time during sensitization (matrix M-3)

The analysis confirmed that incorrect dosing of components has a significant impact on the density of the tested bulk emulsion explosive. In fact, a decrease in density during the first 60 min will result in a VOD increase. However, as shown on the last line of Table 2, a density below 0.8 g/cm^3 could translate into lower VODs. Therefore, the periodic calibration of the mixing-charging units is such an important factor, and has a direct impact on the detonation properties of emulsion explosives, and thus on the effectiveness of the blasting operations.

5 Discussion of Results

This study has confirmed that the sensitizer content has a significant effect on the density of a bulk emulsion explosive and that there are changes in density over time from the moment of sensitization. According to the EU-type examination certificate issued by a notified body, the density of the Emulinit explosive should be between 0.80 and 1.25 g/cm^3 . However, according to the universal instructions for sensitization process control for this type of explosive at a nominal sensitizer content of 4.5% , the density after 30 min at $25 \text{ }^\circ\text{C}$ should range from 0.95 to 1.25 g/cm^3 . It should therefore be assumed that values below 0.95 g/cm^3 are defined as incorrect. Furthermore, the certificate also states

that this explosive may be stored in blastholes for up to 48 h. For technological and organizational reasons, explosives in blastholes in Polish copper mines can be fired from 30 min to 48 h after loading. It can, therefore, be assumed, that not all of the charges are fired at optimal densities.

Consequently, the densities of all of the samples were measured again after 24 h and then after 48 h. The samples were stored in a room with a stable temperature of 25 °C. The results are presented only for the measurements after 24 h, as no further changes in the volumes of the explosive samples were observed after this time.

The density values measured after 24 h indicated that the considered explosive is very sensitive to the passage of time. Reducing the sensitizer dose by 50%, *i.e.* to 2.25% by mass, resulted in an average density increase of 0.29 g/cm³ (in relation to the initial value) to 1.02 g/cm³. Such a density should not affect the detonation parameters of the explosive. On the other hand, increasing the sensitizer content led to a decrease in density to a value below the acceptable level. The actual values were 0.71 g/cm³ for 5.85% of sensitizer and 0.65 g/cm³ for 6.75%. In principle, such a low-density of a bulk emulsion explosive is characterised by a very high sensitivity to initiation, but they are less energetic than high-density ones. This is usually accompanied by a lower VOD and low energetic content by volume. Furthermore, it should be noted that an incorrect density was also observed for a standard sensitizer dose after 24 h, which was 0.79 g/cm³. Certainly, such a low density can only be achieved under laboratory conditions, when the blending of the components is very precise. Mixing of components using charging units in mines is not as precise as manual mixing, hence the nominal content of the sensitizer will not effect such a significant decrease in density. This is because during the mechanical charging, the flow of components in the loading hose is laminar and mixing takes place using a cross-stream static mixer. Due to large differences in the viscosity of both components, the fast flow and relatively short mixing time limited by the length of the static mixer (optimized pressures in the loading hose), complete mixing is not possible, compared with manual mixing under laboratory conditions. Obviously, the density obtained does not have to influence the detonation capacity of the explosive, but it may significantly influence its detonation parameters.

6 Conclusions

- ◆ The results of this experimental research on the impact of sensitizer content on the density of a bulk emulsion explosive, carried out under laboratory conditions, have confirmed that there is a significant relationship between these two parameters. The sensitizer content affects both the final density value (complete conversion of components) and changes in density over time from the moment of sensitization. Reducing the sensitizer content below the recommended level results in a higher density, which is related to a lower amount of lubricant coating on the inner surface of the loading hose. From a mining perspective, this makes proper mixing of the components and the pumping of explosives into the blastholes more difficult, and has a negative impact on the operational parameters of emulsion explosives.
- ◆ Analysis confirmed that increasing the concentration of the sensitizing agent by 30% and 50% led to density reductions of 4% and 7%, respectively. In turn, reducing the sensitizer content by the same percentages resulted in increases in the final density by 7% and 8%, respectively. Bulk emulsion explosives with densities outside the recommended range may exhibit different detonation parameters compared to the declared ones, which in turn may affect the effectiveness of blasting operations.
- ◆ Retaining the optimal emulsion densities in mines until firing in blastholes is a key factor directly influencing the efficiency of blasting.
- ◆ From the perspective of mining operations, the following significant factors should therefore be taken into consideration:
 - verifying the gassing reaction according to instructions before charging of blastholes,
 - controlling the sensitizer content in the explosive and maintaining it at the recommended level,
 - firing of faces in the shortest possible time after charging (considering the recommended 30 min) in order to ensure the maximum efficiency of blasting,
 - training and verification of knowledge and skill of the mixing-charging units' operators, which seems to be one of the most important factors.

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