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FLOTATION KINETICS OF ORTHO-COKING COAL AT DIFFERENT TEMPERATURES

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Abstract: This study investigated the effect of temperature (15; 25; 35°C) on the kinetics and flotation efficiency of hard (ortho-coking) coal using a fixed dose of machine oil as a collector and α-terpineol as a frother. The results show that coal flotation occurs fastest and most efficiently at 15°C, achieving a volatile fraction recovery of 77%. At 35°C, the recovery of volatile parts was 66%, or 47% at 25°C. At the same time, hard coal flotation at 15°C was found to reach a recovery of 45% after just five minutes, almost four times faster than flotation at 25°C.

Keywords: *ortho-coking coal, flotation, flotation kinetics, flotation temperature*

1. INTRODUCTION

Historically, hard coal has been used as one of the most important energy resources in Poland and the European Union (EU). In the second decade of the 20th century, fossil fuels accounted for almost 50% of the energy market within the EU (Ruszel, 2017), as illustrated in Figure 1a. In 2023, the situation has already changed significantly and the share of fossil fuels in the EU market has been reduced to 25% (Figure 1b.).

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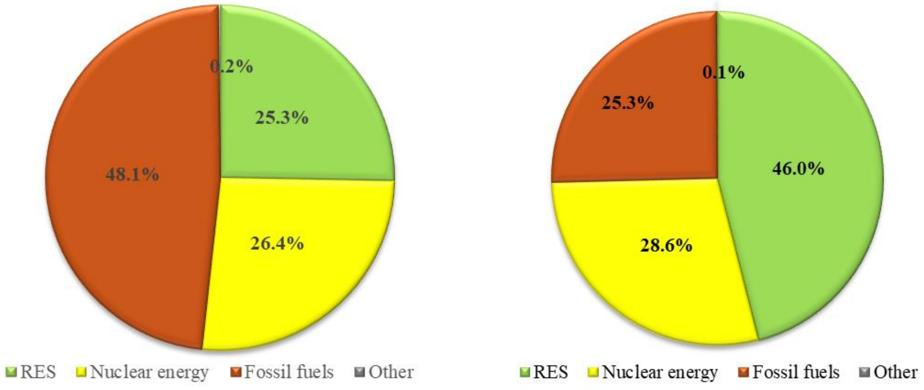
This indicates a shift from fossil fuels as an energy resource, which is in line with current EU policy (Energy Transition Commission, 2025). Coking coal is a strategic and critical raw material and is used as a chemical raw material, e.g. for steel production. (Holappa, 2021). The coal used in steel production must first be refined through the coking process. In Poland, coking coals are mined primarily in the Upper Silesian Coal Basin (Dreger et al., 2024).

One of the main disadvantages of hard coal is impurities. These are mainly connected to coal mining technology. The main reason is the way the machinery used to exploit the coal works: the plow and the shearer. Both machines have a fixed pre-set cutting height. As a result, they are unable to account for the presence of waste rock in the longwall face during extraction, which occurs due to rock mass deformation and various intergrowths (Ostrihansky, 1996). Consequently, the machine extracts both the waste rock and coal, which are mixed in transport and as a result of pre-crushing (Ostrihansky, 1996). Therefore, the coal used for steelmaking needs to first be refined. It is worth noting that ortho-coking coal enriched by flotation is directly used in subsequent stages of steel production, such as coking and the blast furnace process. In the coking process, the coal is heated in the absence of air to temperatures of 1000–1200°C, which leads to degassing and the formation of metallurgical coke. The quality of the coke — including its ash content, reactivity, and mechanical strength — depends largely on the degree of coal purification, which is why flotation is a key stage in feedstock preparation. The resulting coke is then fed into a blast furnace, where it performs three essential functions: it serves as a fuel providing energy, a reducing agent for iron oxides, and a structural component that ensures the permeability of the burden. Consequently, changes in flotation parameters, which affect coal quality and the content of non-combustible matter, have a direct impact on blast furnace efficiency, slag generation, and the overall cost of pig iron production. One of the processes used to enrich coal for fractions smaller than 0.15 mm is flotation (Honaker et al., 2023).



(a) (b)

Fig. 1. Energy sources in the European Union states in 2017 (a) and in 2023 (b) (Ruszel, 2017; Eurostat, 2025).

During flotation, the hydrophobic part of the solid in the aqueous solution combines with a gas bubble, such as air, and floats towards the surface of the liquid (Drzymala, 2009). It is one of the most versatile and all-round enrichment or separation methods, quite simple in terms of the process itself, and yet many improvements are still being made. The reason for this is the high number of parameters that can be controlled in flotation, as indeed in all mineral processing. In the case of flotation, the simplest parameters to change are the amount and concentration of reagents used in the process, the amount of air fed, the grain size of the sample, the number of rotor revolutions and the temperature of the flotation liquid (Drzymala, 2009). By reviewing data from literature, it is apparent that such research is still continuing (Ao et al., 2024; Li et al., 2024; Hasanizadeh et al., 2024; Ren et al., 2023; Qu et al., 2024). In the works carried out at the Wrocław University of Technology, involving copper-bearing shale, flotation was shown to proceed faster and more efficiently for some frothers from the alkyl polyglycol compound group at 15°C (Kaczmarska, Bakalarz, 2016). Different results were observed with coal flotation. Flotation of coal using paraffin and paraffin mixtures did not show any effect of temperature on yield or flotation kinetics (Gyle, Eddy, 1961), while flotation using aliphatic alcohols produced lower carbon yields at higher temperatures (Gyle, Eddy, 1961).

In an engineering approach, the flotation process can be described using chemical reaction kinetics. The reactants are mineral or rock particles and gas bubbles, and the product is the resulting aggregate particle. The flotation rate here will depend on several factors: the number of particles and bubbles, the frequency of their collisions, the stability of the bond after the bubble and particle collide, and the persistence of the bond in the aggregate (Wills, Finch, 2016; Sokolović et al., 2025). The kinetics of flotation is most easily determined using kinetics curves, i.e. a trend line, plotted using the appropriate equations, based on the recovery of the use-ful mineral or concentrate recovery over time (Laskowski, 1989). This relationship is described by the differential equation (1) (Zuniga, 1935):

$$dC/dt = -kC^n, \tag{1}$$

Where:
$C(t)$ - concentration of floatable grains remaining in the flotation cell at time t,
k - flotation speed constant,
n - constant characterising the order of the process (order of process kinetics).

From equation (1) we can derive the flotation kinetics equations, which are used to plot flotation kinetics curves (Brożek, Młynarczykowska, 2009; Drzymala et al., 2017; Drzymala et al., 2022; Sokolović, Mišković, 2018). They are used to get the best possible fit of the curve to the resulting points on the graph. The first-order flotation kinetics equation (2) is most commonly used (Wills, Finch, 2016; Laskowski, 1989):

$$\varepsilon(t) = \varepsilon_{max} \cdot \left(1 - e^{-kt}\right), \tag{2}$$

Where:
ε_{max} - maximum recovery of flotation, %
k - flotation rate constant, min^{-1}
t - flotation time, min.

By plotting flotation kinetics curves as a function of recovery against flotation time for the data obtained, we can determine the quality of flotation. A comparison of example flotation kinetics can be found in Figure 2. Flotation 1 was faster and more efficient where compared to Flotation 2. Faster because it achieved maximum recovery in a shorter time interval. More efficient because it achieved a higher ε_{max} than Flotation 2 (Ratajczak, Drzymała, 2003).

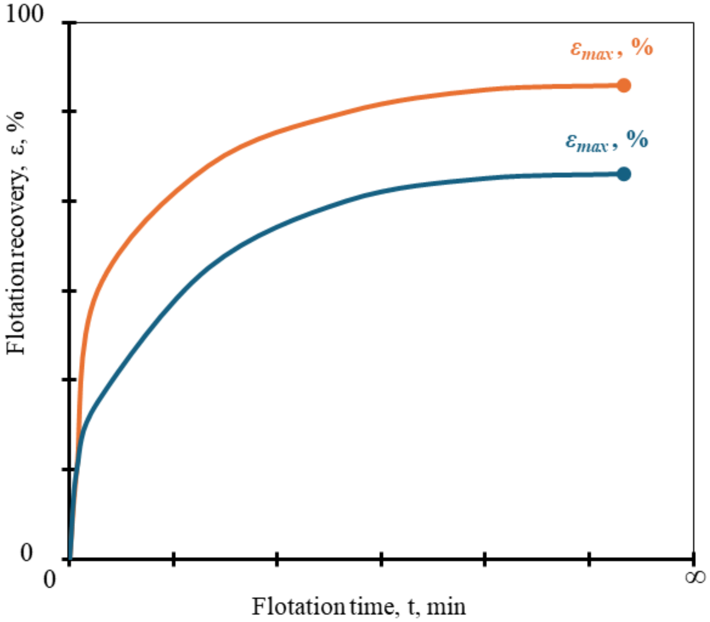


Fig. 2. Examples of flotation kinetics curves (Ratajczak, Drzymała, 2003).

The flotation temperature is one of the basic parameters that can be controlled in flotation. Consequently, a number of studies have been conducted to determine its impact for selected raw materials and reagents, and while all involve temperature, the methodology of the studies can vary widely. For example, studies have been carried out to determine only the effect of temperature on the performance of the collector

(Brożek, Młynarczykowska, 2009). A mixture of quartz particles and glass spheres of different sizes was there used as the flotation material, and dodecylamine hydrochloride (DDAHCl) was used as the collector. Experimental results showed improved flotation kinetics in the 30-40°C temperature range for small particles (Lazarov et al., 1994). Some of the more recent studies have been conducted on rare earth metals, on bastnazite and monazite, with the additional use of hydroxamic acid as a modifier (Lazarov et al., 1994). It was demonstrated that an increase in process temperature leads to higher recovery and a greater content of the valuable component in the concentrate, which is further enhanced by the presence of acid that improves flotation selectivity (Li, 2018). Using copper-bearing shale as an example, where flotation was carried out in a temperature range of 15-35°C, using different glycols as frothers, it was shown that the fastest flotation, with the highest yield, occurred for diethylene glycol monobutyl ether at 15°C, and for most of the other reagents tested, it occurred best at the lowest temperature (Kaczmarska, Bakalarz, 2016).

The used in the investigation coal was rich in carbonaceous matter and the flotation feed and concentrate contained only about 2.5% ash. There is, of course, the possibility of further flotation improvement to increase recovery of coal but this would require additional operations such as grinding and reflotation, possibly at low temperatures and with application of stronger collectors, and probably other modifiers, since, as mentioned in the theoretical section, carbon starts to lose its hydrophobic properties when is very highly carbonized.

By analysing Figures 5 and 6, it is clear that flotation at 25°C has the weakest kinetics among the set temperature range ($K_m = 0.098$ min^{-1}). The averaged cumulative recovery of the combustible substance in the concentrate did not exceed 50%. Flotation had the best kinetics at 15°C, where the averaged cumulative yield was almost 80% ($K_m = 0.192$ min^{-1}). Flotation at 35°C also had good kinetics, with yields of almost 70% ($K_m = 0.111$ min^{-1}). The differences in the flotation kinetics at room temperature (25°C) and at 15°C are noteworthy.

Published literature data on flotation of coals at different temperatures indicate that there is a specific response of each coal. Starting at temperature about 15°C the kinetics or recovery can be better, worse or the same with temperature increase (Bhattacharya, Pascoe, 2005). However, usually around 25°C - 30°C there is either minimum or maximum of flotation. Similar influence of temperature on ortho-coking flotation was obtained for tests involving copper-bearing shale ore investigated in the presence of different foaming agents. There, the copper-bearing shale also floated much better at 15°C for four of the six froths tested (Kaczmarska, Bakalarz, 2016).

Summarising the data, it is clear that the slowest flotation of the investigated ortho-coking coal occurs at room temperature and it would be profitable to consider changing the temperature in the plant flotation halls.

In the case of hard coal, the effect of oxidation degree coal on the floatability was investigated. In the study, coal was subjected to oxidation for a set amount of time at a high temperature. Studies then showed that coals oxidised at higher temperatures flotate better than at lower temperatures (Gyle, Eddy, 1961). Other studies conducted in the 5-75°C temperature range have shown much better flotation at 5°C and 25°C (Hacifazlioglu, 2016).

The aim of this study was to investigate the effect of temperature on the kinetics and performance of hard coal flotation using a constant dose of machine oil as a collector and α-terpineol as a frother, while maintaining constant temperatures in the flotation machine cell. The tests were carried out on a sample of hard ortho-coking coal, type 35.2B.

In paper, it was shown that differences in flotation temperatures of hard coal caused changes of ε_{max} and k. A comparison of flotation kinetics at different temperatures was presented in a graphical form and a modified flotation rate constant K_m (min^{-1}) was used as an alternative to comparing the general flotation reactions, according to the formula (3) (Yang et al., 2021; Bu et al., 2020; Xu, 1998):

$$K_m = \frac{\varepsilon_{max} \cdot k}{100}, \tag{3}$$

Where:
ε_{max}- maximum recovery of flotation, %
k - flotation rate constant, min^{-1}

2. RESEARCH METHODOLOGY

The study used ortho-coking coal, type 35.2B, obtained from the Laboratory of Mineral Processing of the Faculty of Geoengineering, Mining and Geology of Wrocław University of Science and Technology, according to records: CAS 114. The sample tested originated from the JAS-MOS Mine in Poland is a typical coking coal with medium volatile content and good coking properties, mainly used for metallurgical coke production (Szpyrka, 2019).

The test material of 2 kg of coal was first crushed in a jaw crusher to a fraction below 1 mm and then ground in a drum mill to a fraction of 0-0.25 mm. The coal was then quartered to obtain flotation samples of approximately 120 g each. Flotation tests were carried out in a Mechanobr flotation machine. Coal flotation was carried out in a 0.5 dm^3 flotation cell in the presence of 150g/Mg frother and 300g/Mg collector, at temperatures of 15°C, 25°C and 35°C. In order to make the results as accurate as possible, two flotations were carried out for each set temperature and the results from both were averaged.

The temperature was measured in a flotation cell filled with a suspension of aqueous solution and coal at the start and end of flotation. Flotation was to be terminated when the froth had cleared or the temperature of the liquid had dropped by more than 2.0°C. Flotation was carried out at a rotor speed of 2000 rpm.

The product remaining in the chamber was treated as waste. The products, collected in separate containers after froth suppression and decantation, were placed in a dryer for drying. After drying, the products were weighed to the nearest 0.1 g to determine their yields. Each product was subsampled to 1 g, placed in a porcelain crucible, and weighed with an accuracy of 0.0001 g. The crucibles containing the samples were placed in a muffle furnace preheated to 300°C. Once the furnace reached 800°C, the samples were ashed for 2 hours at that temperature. After ashing, the crucibles were reweighed to determine the mass of the remaining ash, thus allowing the ash content of each product to be calculated. A more detailed description of the research methodology can be found in Dykiert (Dykiert, 2019).

3. RESULTS AND DISCUSSION

Table 1 shows the results of weighing the products of the individual flotations and their ashes. Based on the data obtained, the ash content of the individual products was calculated.

The investigated coal contained approximately 97% carbonaceous matter, which also reflects the type of coal used in the study. This is reflected in the graph as a Mayer curve showing the relationship between the recovery of the valuable product and the yield (Figure 3). Figure 3 shows that the selectivity of separating carbonaceous matter from ash is low due to its low carbon content, making it highly energetic.

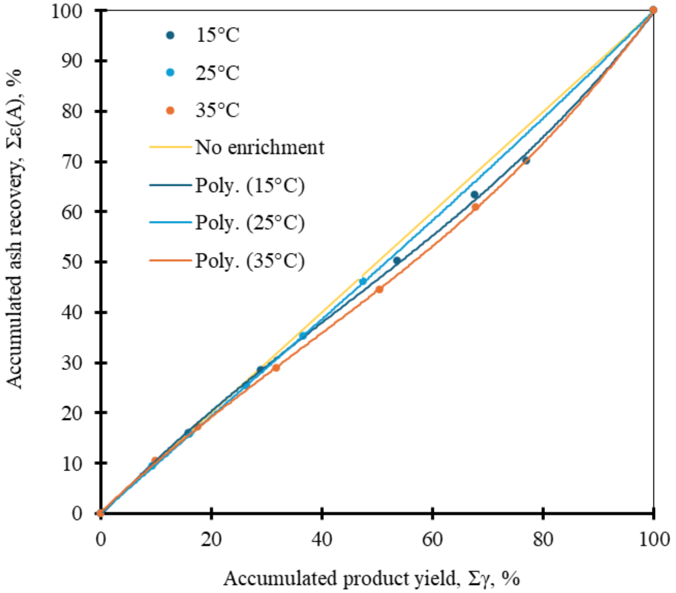
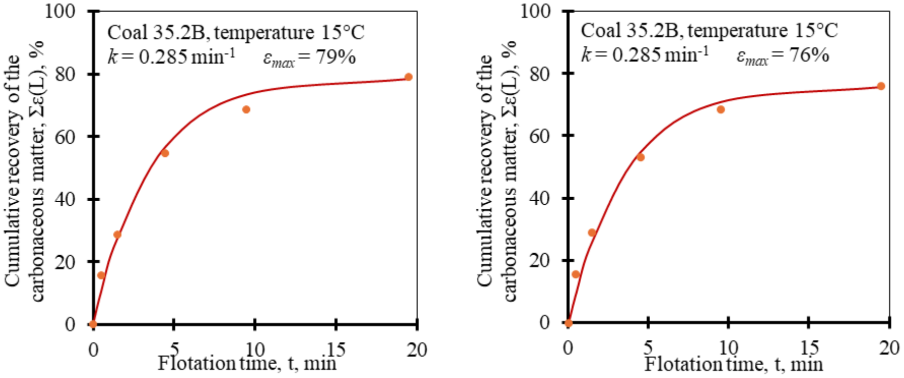


Fig. 3. Enrichment of hard coal at 15 °C, 25 °C, 35 °C in the Mayer curve system.

Figure 4 shows the kinetics of all flotations, i.e. the recovery value of the volatile parts of the tested coal over time. A trend line was fitted to the measurement points obtained using the first-order kinetic equation (Table 1) and the obtained parameter values for the flotation rate constant *k* and the maximum recovery ε_{max}.



(a) (b)

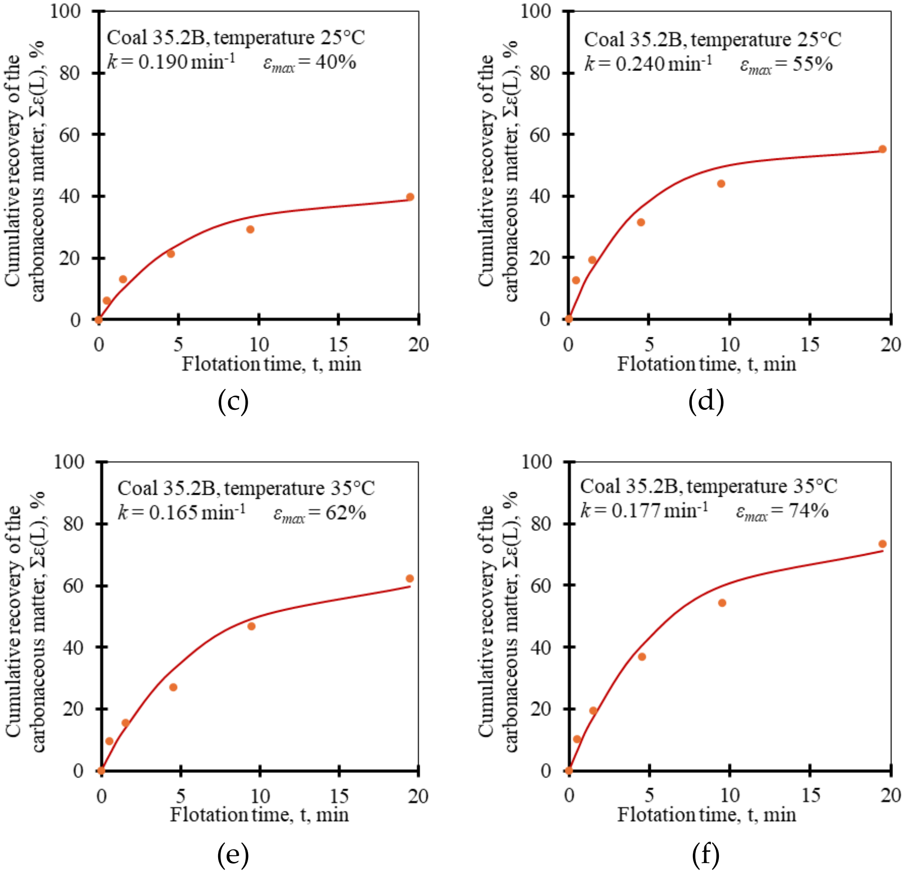


Fig. 4. Flotation kinetics of hard coal at temperatures (a, b) 15°C, (c, d) 25°C and (e, f) 35°C

Table 1. Yields of hard coal and ash.

	Flotation number	**1a**		**1b**	
Flotation of coal at 15°C	**Type of product weighed**	**coal sample, g**	**ash, g**	**coal sample, g**	**ash, g**
	Product K1	0.8722	0.0224	0.9542	0.0239
	Product K2	1.0551	0.0270	1.1532	0.0270
	Product K3	1.3375	0.0311	1.2764	0.0281
	Product K4	1.0868	0.0251	1.0053	0.0241
	Product K5	1.2440	0.0228	1.0835	0.0206

	Waste	1.4641	0.0518	0.9452	0.0293
Flotation of coal at 25°C	**Flotation number**	**2 a**		**2b**	
	Type of product weighed	**coal sample, g**	**ash, g**	**coal sample, g**	**ash, g**
	Product K1	0.8994	0.0230	0.8441	0.0198
	Product K2	0.8726	0.0209	0.8703	0.0193
	Product K3	1.0988	0.0280	0.9147	0.0196
	Product K4	0.8784	0.0229	0.8854	0.0196
	Product K5	1.0775	0.0283	0.9826	0.0221
	Waste	0.9685	0.0244	1.1421	0.0290
Flotation of coal at 35°C	**Flotation number**	**3 a**		**3b**	
	Type of product weighed	**coal sample, g**	**ash, g**	**coal sample, g**	**ash, g**
	Product K1	1.0246	0.0262	1.1614	0.0327
	Product K2	1.1205	0.0255	1.0801	0.0241
	Product K3	0.9656	0.0205	1.0699	0.0219
	Product K4	0.9546	0.0201	1.0984	0.0237
	Product K5	0.9031	0.0245	1.1287	0.0242
	Waste	1.1823	0.0319	1.3252	0.0479

Figure 5 shows the averaged flotation kinetics curves of the test coal for each of the investigated temperatures. As can be seen from the kinetics curves, the fastest and most efficient flotation occurred at 15°C, where the highest recoveries were achieved most quickly, ending with a cumulative highest recovery of approximately 77%. Flotation at 35°C proceeded less efficiently and rapidly, achieving a cumulative recovery of 66%. The weakest and slowest flotation was observed at 25°C, where a maximum cumulative recovery of 47% was achieved.

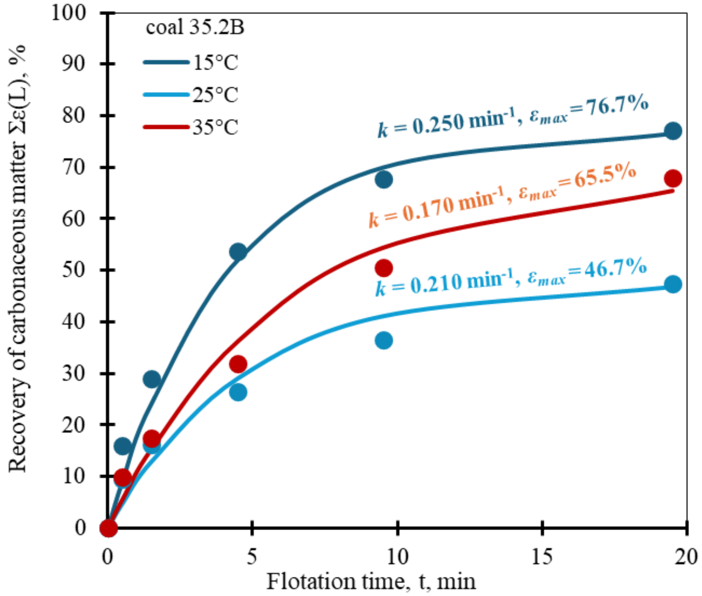


Fig. 5. Hard coal flotation kinetics in relation to temperature (averaged values).

In addition, Figure 6 shows the dependence of the maximum recovery on the temperature of coal flotation. For each temperature, the results from both trials (Table 1) were averaged. The best maximum recovery was achieved at 15°C, the second-best result at 35°C and the worst result at 25°C. From this, it is possible to formulate the relationship that the recovery initially decreases with increasing temperature and then starts to increase from 25°C onwards.

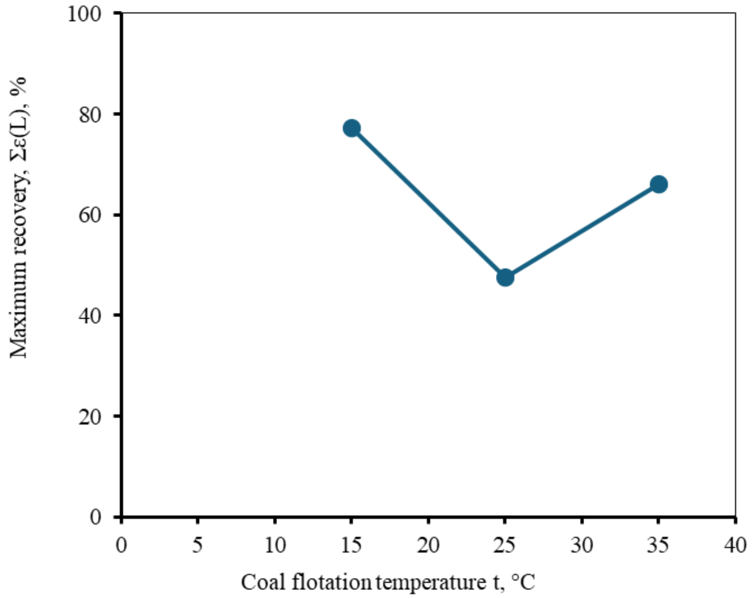


Fig. 6. Dependence of maximum recovery of hard coal on flotation temperature in the presence of α-terpineol and machine oil.

4. SUMMARY

Using the example of the samples obtained, effect of temperature on the enrichment of the hard coal in was observed, confirming its high degree of carbonization. Very good coal enrichment results were obtained for the adopted test methodology, yielding high volatile matter contents of 97.4-97.6% and thus ash contents in the range of 2.4-2.6%. There is, of course, the possibility of further enrichment of the coal but this would require flotation to be carried out over a very long period of time, which is incompatible with the assumptions of the study because, as time passes, the temperature in the cell becomes more and more equalised with that of the environment, thus deviating from the assumptions of the work. In addition, this would require the use of strong collectors and probably other modifiers, since, as mentioned in the theoretical section, carbon starts to lose its hydrophobic properties when very highly carbonized.

By analysing Figures 5 and 6, it is clear that flotation at 25°C has the weakest kinetics among the set temperature ranges ($K_m = 0.098$ min^{-1}). The averaged cumulative recovery of the combustible substance in the concentrate did not exceed 50%. Flotation had the best kinetics at 15°C, where the averaged cumulative yield was almost 80% ($K_m = 0.192$ min^{-1}). Flotation at 35°C also had good kinetics, with yields of almost 70% (K_m = 0.111 min^{-1}). The differences in the flotation kinetics at room temperature are noteworthy, with cumulative recoveries of 55% and 40%. This is a large difference and is most likely due to the heterogeneity of the samples. The quartering method applied to reduce the 2-kilogram coal sample to 120-gram test portions is not without limitations. Although the carbon content in the feed was relatively consistent (2.4% and 2.5%, respectively), variations in ash composition may have occurred, potentially influencing the flotation kinetics.

Similar results were obtained for tests on copper-bearing shale in the presence of different foaming agents. There, the copper-bearing shale also flotated much better at 15°C for four of the six froths tested (Kaczmarska, Bakalarz, 2016).

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