Influence of N-Alkanes on Adhesion of an Air Bubble to the Surface of Low-Rank Coals

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Measurements of the detachment force of an air bubble from the surface of coals of the ranks: 31.1, 31.2, 32.1 and 32.2 were carried out. The coal surface was precovered with n-alkane film in the homologous series from n-hexane to n-hexadecane. The forces were compared with those calculated theoretically on the basis of the previously determined values of the surface free energy components and of the contact angles measured in a coal/n-alkane film — air bubble — water system. On the basis of the conducted measurements and calculations we confirmed that the stability of the coal/n-alkane film — air bubble — water systems depends on the rank of the coal studied, the thickness and kind of the hydrocarbon film present on the coal surface, and on the water film pressure under the air bubble. We also confirmed that the stability of n-alkane films on the surface of the coals studied is smaller than the stability of these films on the surface of typical hydrophobic solids.

By comparison of the measured and calculated stabilities of the coal/n-alkane film — air bubble — water system it appeared that the stabilities of such systems may be predicted on the basis of the contact angle of the coal/n-alkane film — air bubble — water system and of the dispersion and nondispersion components of the surface free energy of coal.

INTRODUCTION

A big demand for all kinds of minerals and ores forces us to exploit increasingly poorer minerals, which must be beneficiated before use. Among the methods commonly applied to enrich these minerals, froth flotation can be mentioned. The method is very useful in recovering coal fines.[1-3]

The amount of coal fines increases markedly after introduction of modern mining operations, which may sometimes generate more than 25% of coal fines.

A selective mineralization of air bubbles is one of the main problems in the flotation process, and the efectiveness of this process depends, among other things, on the kind and composition of the flotation reagent, size of air bubbles and size and kind of the grains floated.

The force necessary to disrupt an air bubble from a solid surface may be regarded as the mineralization measure of an air bubble. This does not only

depend on the size of grains and air bubbles but also on the components of the surface free energy of the solid and water.[4-6]

Addition of a flotation reagent into the flotation pulp makes the contact of an air bubble with grains of the floated mineral easier as a result of the creation of a mixed film on their surface, changing both the components and the total surface free energy of a solid.[5,7]

In coal flotation reagents such as kerosene or fuel oil which contain the saturated hydrocarbons are used as collectors. Accordingly, the knowledge of influence of the n-alkanes on the adhesion of an air bubble to the coal surface may be useful for a better understanding of the attachment of bubbles in froth flotation of coal.

In order to understand this influence, measurements of the detachment force of an air bubble from the coal surface precovered with n-alkanes in the homologous series from n-hexane to n-hexadecane were carried out. Moreover, a theoretical analysis of the equilibrium state of the coal/n-alkane film — air bubble — water system was also performed on the basis of the previously determined components of the surface free energy of the coals studied.[8]

EXPERIMENTAL

The samples of coal used to examine the stability of coal/n-alkane film-air bubble-water systems originated from the Polish colliers of Siersza, Jankowice, Gotwald and Kleofas, the ranks of which, according to the Polish classification,[9] were: 31.1, 31.2, 32.1 and 32.2, respectively. Some characteristic properties of the coals used in these studies are listed in Table I.

TABLE I

Ranks of Coal According to the Polish Classification and Some of Their Properties

Rank of coal	Volatile matter (5)	properties Ability of caking (LR[a])	Caloric value J/kg × 10⁶
1. Flaming coal-31.1	>30	0—10	28.470—30.560
2. Flaming coal-31.2	>30	0—10	30.560—32.657
3. Gas flaming coal-32.1	>30	10—35	32.657—33.913
4. Gas flaming coal-32.2	>30	10—35	>33.913

[a] — LR — Roga number

Pieces of coal of the above mentioned ranks were carefully selected under the microscope, excluding those with cracks, mineral substances, different macerals, particles of pyrite, occlusions etc. The most regular pieces of coal selected were stored for a few months in a desiccator filled with molecular sieves of two sizes (4Å + 5Å), to protect them against water adsorption.

Then, the pieces were polished parallel to the bedding plane using the method described earlier.[8] From the obtained coal plates, very thin lamellae with one polished side were split off ond cut into small grains, 2 × 2 mm in size. These grains were attached using paraffin to a quartz rod[5] calibrated by measuring the deviation of the rod under the well-known weight with a cathetometer. To form the n-alkane film on the surface of the grain, it was immersed in the hydrocarbons studied. The grain was taken out and the excess of hydrocarbon was removed by tender shaking and then it was washed several times with doubly distilled water. The quartz rod with the attached coal grain

was connected to a measurement device and immersed in doubly distilled water in a measuring vessel. Next, the coal grain was contacted with an air bubble of 4.05 mm in diameter which was pushed out of a capillary by means of a micro-meter screw. The detachment force was measured as described earlier.[10,11] In this method the values of the detachment force were read out under the microscope from the deviation of the quartz rod. After that, the air bubble was removed and a new one of the same size was pushed from the capillary and contacted with the same grain surface and the detachment force was measured again. This measurement procedure was repeated until a constant value of the detachment force was obtained. Then, the air bubbles were contacted with and disrupted from the grain surface without measuring the detachment force. Only the detachment force for the hundredth air bubble attached to the grain surface was measured again.

The measurements of the detachment force were made in the same way for all the studied ranks of coal, precovered with various hydrocarbons in the homo-logous series from n-hexane to n-hexadecane. For any given coal/n-alkane-film — air bubble — water system the detachment force was measured for at least ten coal grains.

All measurements were carried out at $20\,^{\circ}C \pm 0.1\,^{\circ}C$ and the sensitivity of the method was 5×10^{-4} mN.

RESULTS AND DISCUSSION

The force required for the detachment of an air bubble from the surface of all ranks of coal wetted with the shorter chain of n-alkanes (n-hexane, n-heptane and n-octane) decreased with the increase of the number of the disrupted air bubbles until the characteristic constant value for a given system was reached. In the case of the surface of coal grains which were precovered with n-alkanes in the homologous series from n-nonane to n-hexa-decane, the detachment force increased at first to a maximum value and then decreased to the characteristic constant value when the number of disrupted air bubbles increased.

In Figure 1, for example, the changes of the detachment force of an air bubble from coal rank 31.1, wetted with n-hexane (curve 1), n-undecane (curve 2) and n-hexadecane (curve 3), are presented as a function of the number of disrupted air bubbles. The same relationship for coal rank 32.2 is shown in Figure 2.

It can be seen from Figure 1 that for coal rank 31.1, wetted with n-hexane (curve 1), the stability of the coal/n-hexane film — air buble — water system decreases slightly during disruption of the first three air bubbles and the disruption of the following air bubbles does not significantly affect the detachment force (F_o).

In the case of coal of the same rank but wetted with n-undecane (curve 2) or hexadecane (curve 3) for the first three air bubbles disrupted from this surface the detachment force increases to a maximum and then decreases until the 17-th air bubble is disrupted.

The detachment force of air bubbles from the 32.2 coal rank (Figure 2), wetted with n-hexane (curve 1), n-undecane (curve 2) and n-hexadecane (curve 3), changes in the same way as for coal rank 31.1 (Figure 1). However, to obtain constant values of the detachment force in the above cases, a much greater number of air bubbles must be disrupted from the coal surface wetted with n-alkanes. The maximal values of the detachment force for coal rank 32.2, precovered with a film of n-undecane or n-hexadecane, were
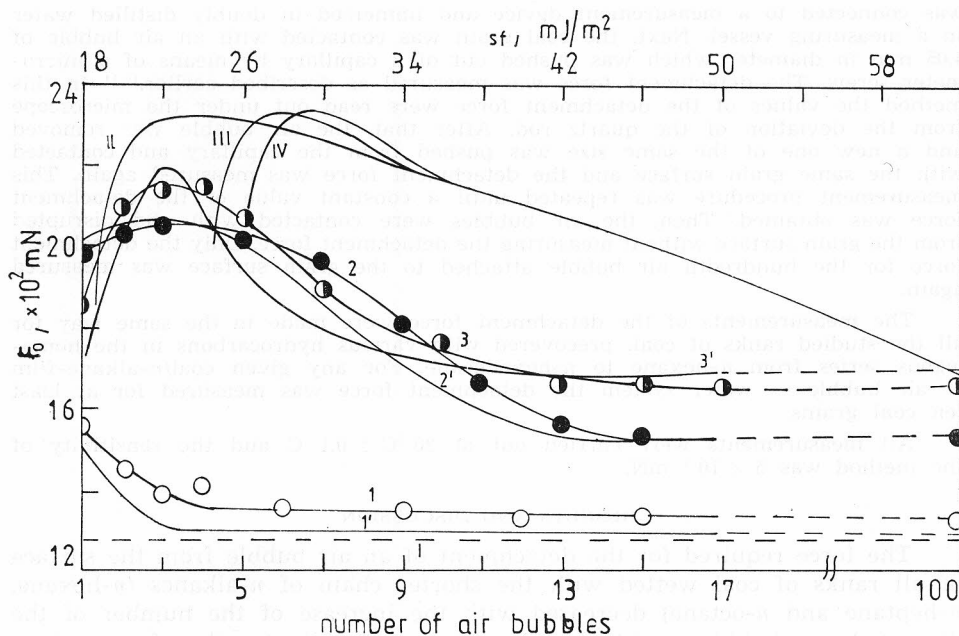
Figure 1. The relationship between the detachment force of an air bubble from the surface of coal rank 31.1 and the number of disrupted air bubbles. Curves 1 (—O), 2 (—●) and 3 (—◐) show the measured values, while curves 1′, 2′ and 3′ the values, calculated from Eq. (3), of the detachment force of an air bubble from coal surface precovered with n-hexane film (curves 1 and 1′), n-undecane (curves 2 and 2′ and n-hexadecane (curves 3 and 3′). Curves II, III, and IV show the changes of the values of $F_{o(max)e}$ as a function of γ'_{sf} (upper scale) from the coal surface precovered with a film of n-hexane (curve II), n-undecane (curve III) and n-hexadecane (curve IV). Dotted line shows the value of the detachment force of an air bubble from the »bare« coal surface.

obtained only when a much greater number of air bubbles were disrupted than for similarly treated samples of coal rank 31.1 (Figure 1).

On the basis of the results presented in Figures 1. and 2. we may state that, to obtain a constant value of the detachment force of air bubbles from the coal surface, a larger number of the disrupted air bubbles is needed as the coal rank grows from rank 31.1 to 32.2 and as the length of the hydrocarbon chain increases from n-hexane to n-hexadecane.

It should be emphasized that the dependence of the detachment force on the number of disrupted air bubbles is slightly different when the coal surface is wetted with either odd or even hydrocarbons, so the films of these hydrocarbons must be different in structure; this fact has been confirmed many times[5,11] and is supported by the results presented in Figure 3. In this figure, the relationship between the constant value of the detachment force (F_o) and the number of carbon atoms in hydrocarbon molecules used for wetting coal ranks of 31.1 (curve 1), 31.2 (curve 2), 32.1 (curve 3) and 32.2 (curve 4) is presented. It can be seen that the detachment force increases stepwise with the increase in the number of carbon atoms in the n-alkanes used for precovering the coal surface.
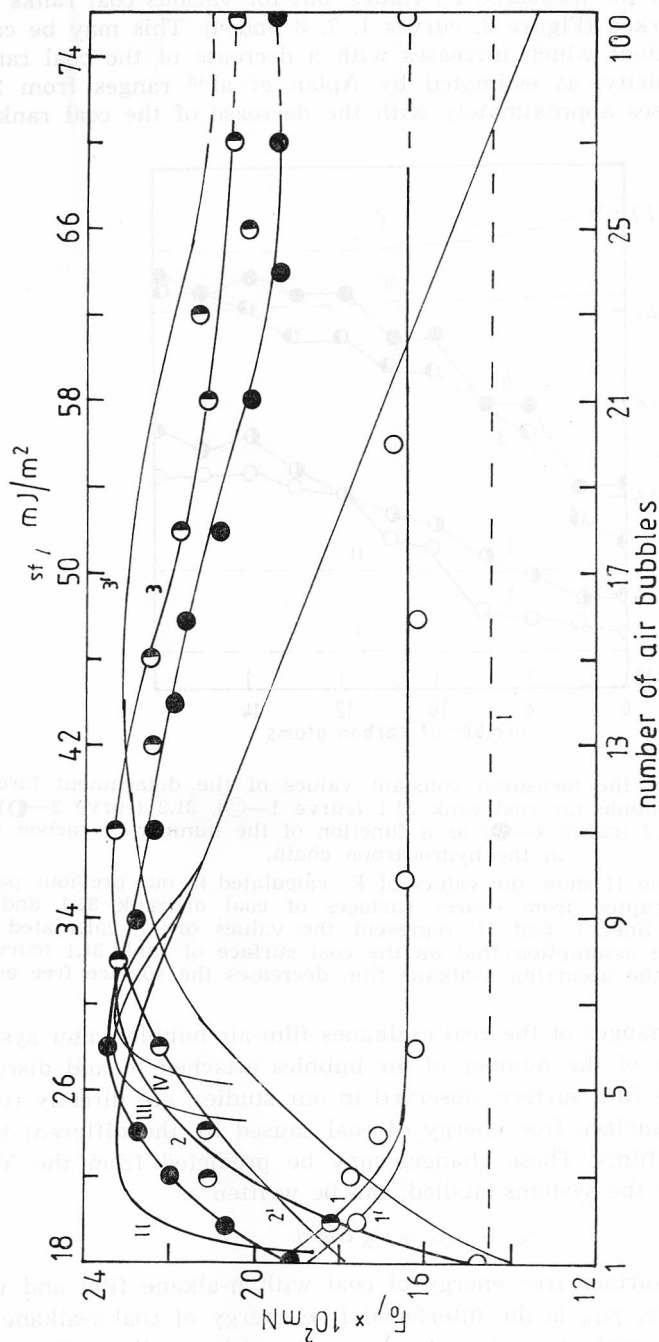
Figure 2. The relationship between the detachment force of an air buble from the surface of coal rank 32.2 and the number of disrupted air bubbles. Curves 1 (—O), 2 (—$\bullet$) and 3 (—$\bullet$) show the measured values while curves 1', 2' and 3' the values, calculated from Eq. (3), of the detachment force of an air bubble from the coal surface precovered with a film of n-hexane (curves 1 and 1'), n-undecane (curves 2 and 2') and n-hexadecane (curves 3 and 3'). Curves II, III and IV show the changes of the values of $F_{o(max)c}$ as a function of γ_{sf} (upper scale) for the coal surface precovered with a film of n-hexane (curve II), n-undecane (curve III) and n-hexadecane (curve IV). Dotted line shows the value of the detachment force of an air bubble from the »bare« coal surface.

For coal belonging to the same rank (31 or 32) there are insignificant differences between the measured F_o values, but for various coal ranks these differences are marked (Figure 3, curves 1, 2, 3 and 4). This may be caused by the oxygen content which increases with a decrease of the coal rank.[3,12] Surface hydrophobicity, as estimated by Aplan et al.[13] ranges from 22 to 71% and it decreases approximately with the decrease of the coal rank.
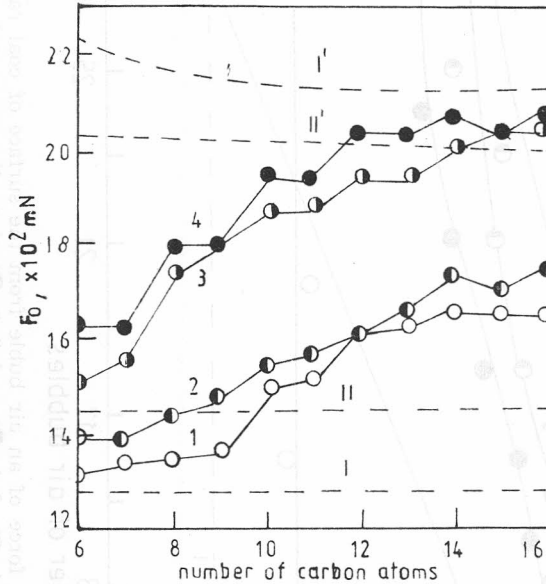

Figure 3. Changes of the measured constant values of the detachment force for the hundredth air bubble for coal rank 31.1 (curve 1—○), 31.2 (curve 2—◑), 32.1 (curve 3—◑) and 32.2 (curve 4—●) as a function of the number of carbon atoms in the hydrocarbon chain.

Dotted lines I and II show the values of F_o, calculated in our previous paper[1], for air bubbles disrupted from »bare« surfaces of coal of rank 31.1 and 32.2, respectively. Dotted lines I' and II' represent the values of F_o, calculated from Eqs. 2 and 3, on the assumption that on the coal surface of rank 31.1 (curve I') and 32.2 (curve II') the occurring n-alkane film decreases the surface free energy.

The stability changes of the coal/n-alkanes film-air bubble-water systems, due to the increase of the number of air bubbles attached to and disrupted from coal/n-alkanes film surface, observed in our studies, are directly related to changes of the surface free energy of coal caused by the different thickness of n-alkanes films. These changes may be predicted from the Young equation which, for the systems studied, can be written as

$$\gamma_{Sf_1} - \gamma_{SfW} = \gamma'_W \cos \Theta \qquad (1)$$

where γ_{Sf_1} is the surface free energy of coal with n-alkane film and water under an air bubble, γ_{SfW} is the interfacial free energy of coal/n-alkane film — water, γ'_W is the surface tension of water reduced by n-alkane film and Θ is the water contact angle.

Assuming that $\gamma_{Sf_1} = \gamma_{Sf} - \pi e$ (where πe is the difference between the surface free energy of coal with a film under the air bubble and the surface free energy with a film in the interfacial region of coal/n-alkane film — water, $\gamma'_W \approx \gamma_W$) and if γ_{SfW} is expressed as the geometric mean from dispersion[14] and nondispersion[15-20] intermolecular interactions, then from Eq. (1) we obtain:

$$\gamma_W \cos \Theta = -\gamma_W + 2 \, (\gamma_{Sf}^d \, \gamma_W^d)^{1/2} + 2 \, (\gamma_{Sf}^n \, \gamma_W^n)^{1/2} - \pi e \tag{2}$$

where γ_W^d and γ_W^n are the dispersion and nondispersion components of the surface tension of water, γ_{Sf}^d and γ_{Sf}^n are the dispersion and nondispersion components respectively, of the surface free energy of coal precovered with a film of n-alkane.

From Eq. (2) the values of the contact angle in the coal/n-alkane film — air bubble — water system may be calculated for a given thickness of n-alkane film. It may be assumed that the surface free energy of coal changes under the influence of n-alkane film which changes with an increase of its thickness from γ_S to γ_H (where γ_S is the surface free energy of coal, γ_H is the surface tension of hydrocarbon) and that in the first place the dispersion component (γ_S^d) of the surface free energy of coal changes to the γ_H value and next the nondispersion component (γ_S^n) drops to zero. Then, we may theoretically determine the changes of Θ values as related to the thickness of n-alkane film in two extreme cases, i. e. when $\pi e = 0$ or $\pi e = \gamma_{Sf} - \gamma_W$.[8,9]

It seems that if the contact angle values measured for the captive air bubble differ only slightly from those measured at the moment of disruption of an air bubble from a small mineral grain surface[4,21], then the maximum values of the detachment force ($F_{o\,(max)\,c}$ may be calculated from the previously derived equation[21]:

$$F_{o\,(max)} = \pi r_{k\,(k)} \, \gamma_W \sin \Theta \tag{3}$$

where $r_{k\,(k)}$ is the radius of critical contact plane[21] at the moment of an air bubble disruption from a large surface of a solid in water. The values of $r_{k\,(k)}$

$$r_{k\,(k)} = \frac{R \sin \Theta}{2} \left[\frac{4}{2 + 3/2 \, (4 - \sin^2 \Theta)^{1/2} - 1/8 \, [(4 - \sin^2 \Theta)^3]^{1/2}} \right]^{1/3} \tag{4}$$

are calculated from the equation[19]:

Using the values of γ_S^d and γ_S^n determined in our previous paper,[8] γ_H taken from the paper[22] (Table II) and $\gamma_W^d = 21.8$ mN/m, $\gamma_W^n = 51$ mN/m and $\gamma_W = 72.8$ mN/m.[14] and assuming that $\pi e = 0$, $F_{o\,(max)\,c}$ values for air bubbles disrupted in water from the 31.1 and 32.2 coal surfaces wetted with the studied n-alkanes were calculated from Eqs. (2), (3) and (4) for γ_{Sf} changing from γ_S to γ_H. $F_{o\,(max)\,c}$ values are shown in Figures 1. and 2. as functions of γ_{Sf} for coal surfaces wetted with n-hexane (curve II), n-undecane (curve III) and n-hexadecane (curve IV).

In these Figures the values of $F_{o\,(max)\,c}$ calculated from Eq. (3) for the previously measured[23] values of the contact angle in coal/n-alkane film — air bubble — water systems are also presented as functions of the number of air bubbles contacted with the studied coal surface precovered with a film of n-hexane (curve 1′) n-undecane (curve 2′) and n-hexadecane (curve 3′)

TABLE II

Literature Values of the Surface Tension of n-Alkanes and the Surface Tension of Water and its Components

n-Alkane	γ_H	n-Alkane	γ_H	γ_W^d	γ_W^n
Hexane	18.49	Dodecane	25.08		
Heptane	20.30	Tridecane	25.38		
Octane	21.80	Tetradecane	25.60		
Nonane	22.91	Pentadecane	25.80		
Decane	23.90	Hexadecane	26.35		
Undecane	24.70	Water	72.30	21.80	51.00

Curves I in Figures 1 and 2 present the changes of $F_{o\,(max)\,d}$ measured for air bubbles disrupted from »bare« coal surfaces of rank 31.1 and 32.2, respectively.[23]

As it can be seen from Figures 1 and 2, the $F_{o\,(max)}$ for the studied hydrocarbons (curve II, III and IV) increase to a maximum value with an increase of γ_{Sf} values to $\gamma_{Sf}^d = \gamma_H$ and $0 < \gamma_{Sf}^n < \gamma_S^n$. Further increase of the γ_{Sf} values (decrease in the thickness of the n-alkane film) causes a decrease of the $F_{o\,(max)\,c}$ values up to those coresponding to $\gamma_{Sf} = \gamma_S$.

It is characteristic that curves II, III and IV become one curve with an increase of γ_{Sf} values. Comparing curves II, III and IV with curves 1, 2 and 3, it may be stated that the shapes cf curves III and IV are similar to curves 2 and 3, but the values of $F_{o\,(max)\,c}$ are distinctly higher; however, there is complete agreement between the values of F_o determined experimentally (Figures 1. and 2., curves 1, 2 and3) and those calculated from Eq. (3) on the basis of the measured contact angles in the coal/n-alkane film — air bubble — water system[23] (Figures 1. and 2., curves 1′, 2′ and 3′). The higher values of the detachment force calculated on the basis of the values of γ_{Sf}^d and γ_{Sf}^n from Eqs. (2) and (3) in comparison with those measured suggest that on the coal surface precoated with n-alkane film (at least with a very thin film) also a water film may be present.[25] In other words, the air bubble contacting with the surface of coal/n-alkane film does not completely remove water from this surface, and the value of πe in Eq. (2) is larger than zero.

The data presented in Figure 3 confirm this fact.

In Figure 3, curve I and II (dotted lines) concerning the values of $F_{o\,(max)}$ calculated from Eq. (3) on the basis of contact angles calculated from Eq. (2) on assumption that $\pi e = \gamma_S - \gamma_W$ for »bare« surfaces of the coal ranks of 31.1 and 32.2, respectively.

For the same coal ranks, curve I′ and II′ refer to the values of $F_{o\,(max)}$ calculated from Eq. (3) on the basis of contact angles calculated from Eq. (2) on assumption that $\gamma_S = \gamma_S^d - W_S + \gamma_S^n$ and $\pi e = 0$.

W_S values equal to the spreading work of hydrocarbon on these coal surfaces were calculated from the equation:[20,26]

$$W_S = 2\,(\gamma_S^d\,\gamma_H^d)^{1/2} - 2\,\gamma_H \tag{5}$$

where $\gamma_H = \gamma_H^d$ [14] is the surface tension of hydrocarbon.

The values of γ_S^d, γ_S^n and γ_H used to calculate W_S were taken from the literature[8,22] (Tables II and III).

TABLE III

Literature Values of the Dispersion and Nondispersion Components of the Surface Free Energy of the Studied Coals and the Adhesion Work of n-alkanes and Water to Coal Surface

Coal ranks	γ_s^d	γ_s^n	$W_{A(H)}$											$W_{A(W)}$
			C_6	C_7	C_8	C_9	C_{10}	C_{11}	C_{12}	C_{13}	C_{14}	C_{15}	C_{16}	
31.1	53.2	8.90	62.73	65.73	68.11	69.82	71.32	72.50	73.05	73.49	73.81	74.10	74.88	110.72
31.2	53.8	8.72	63.08	66.10	68.49	70.22	71.72	72.91	73.47	73.90	74.22	74.81	75.30	110.67
32.1	64.4	9.49	69.01	72.31	74.94	76.82	78.46	79.77	80.34	80.86	81.21	81.52	82.39	118.94
32.2	66.1	9.74	69.92	73.26	75.92	77.83	79.49	80.81	81.43	81.92	82.27	82.59	83.47	120.50

From Figure 3 it is seen that the F_o values measured for the hundredth air bubble disrupted from the surface of coal wetted with n-alkane (rank 31.1 curve 1 and 32.2 curve 4) run between curve I and I' and between curve II and II', respectively.

In the case of coal rank 31.1, the values of detachment forces are close to those calculated from Eq. (3) for the »bare« coal surface. But for coal rank 32.2 the detachment forces from the surface of coal/n-alkane film are closer to those calculated from Eq. (3) for contact angles corresponding to n-alkanes films resulting from the spreading wetting process. In the case of the n-heptane and n-hexane film, the measured values of the detachment force of air bubbles from the surface of coal rank 32.2 are slightly higher than the F_o values calculated for the »bare« coal surface.

On the basis of the data presented in Figure 3 we may state that n-alkane films on the surface of coal ranks 31.1 and 31.2 are considerably less stable than on the surface of coal rank 32.2. In our studies, hydrocarbons were deposited on »dry« surfaces of coals. In the flotation processes, apolar collectors were added into the flotation pulp and they were deposited on the surface of coal grains from the water phase. Thus, based on the above considerations, we may say that coal rank 31.1 should not float if only apolar collectors are used. This fact is confirmed by the coal flotation results[12,27.28] and by the values of the adhesion work of water, n-hexane and of n-hexadecane to the coal surface of ranks 31.1, 31.2 and 32.2 calculated from equations[20,26]:

$$W_{A(W)} = 2 \ (\gamma_S^d \ \gamma_W^d)^{1/2} + 2 \ (\gamma_S^n \ \gamma_W^n)^{1/2} \tag{6}$$

$$W_{A(H)} = 2 \ (\gamma_S^d \ \gamma_H^d)^{1/2} \tag{7}$$

In these calculations the literature values of γ_S^d, $\gamma_S^{n\,8}$, $\gamma_H^d = \gamma_H^{\,22}$ and γ_W^d and $\gamma_W^{n\,14}$ (Tables II and III) were used.

It is seen from these results that for all the systems studied $W_{A(W)} > W_{A(H)}$ and the ratio of $W_{A(W)} : W_{A(H)}$ decreases in the series of the studied coals from ranks 31.1 to 32.2.

This fact points to the conclusion that it is more difficult for apolar liquids from the water phase to contact coal surfaces and that the lower the coal rank the more difficult is the contact of apolar liquids with the coal surface.

On the basis of the experimental and theoretical research we may state that the alkanes strengthen the bond of air bubbles with the coal surface. However, the films of these liquids on coal surface are less stable because the adhesion work of water to coal surface is bigger than the adhesion work of n-alkanes to coal surface. The stability of n-alkane films increases with an increase of the length of hydrocarbon chains and it depends on the kind of hydrocarbon (odd or even). Stepwise changes of the stability of the coal/n-alkanes film — air bubble — water system, as a function of the number of carbon atoms in the n-alkane present on the surface of coal, were recorded.

On the basis of the studies carried out we may assume that a water film is left in the form of clusters under the air bubble in contact with the coal surface precovered with n-alkane films (at least with thin ones) which decrease the stability of coal/n-alkane film — air bubble — water systems.

The conclusion should be confirmed by further studies of the water vapour adsorption on coal surface precovered with n-alkane films of suitable thickness.

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SAŽETAK

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Homologni niz alkana od n-heksana do n-heksadekana je upotrebljen za stvaranje filma na površini ugljena tipa 31.1, 31.2, 32.1 i 32.2. Opisane su sile i značajke dobivenog sustava: ugljen/n-alkanski film—zračni mjehurić—voda.