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## SILICA INTERPARTICLES ACTION IN ALKANES

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Measurements of the destruction time of the sediment column structure of silica particles were carried out for different fractions of silica particles in alkanes from decane to hexadecane. The authors studied the correlations between the measured destruction time of the silica particles sediment column structure and the bulk properties of silica particles and alkanes, as well as the alkane-air, silica particles-air and silica particles-alkane interfacial properties. On the basis of this study linear relationships between the reciprocal of the destruction time and the average diameter of the silica particles fractions, the work of alkanes cohesion, the alkanes density and the difference between the detachment and attachment forces were found. From these relationships, the critical values of the particle diameter, the cohesion work of the alkane density and the difference between the detachment and the attachment forces were determined. It was found that for the systems, having critical values of the parameters mentioned above, the detachment force is equal to the attachment force, both resulting from gravitational and interfacial interactions, respectively. It also results from the present study that the attachment force between silica particles depends on the work of alkane cohesion and the work of silica particle adhesion to liquid, and that destruction of silica particles sediment column structure takes place as a result of interruption of the alkane film between two silica particles. The changes of the destruction time as a function of the number of carbon atoms in the molecule of alkanes occurred as a result of the decreased detachment force and the perimeter of the contact plane and increased attachment force between silica particles in alkanes.

*Key words:* Adhesion, silica particles, alkanes, attachment and detachment forces.

### INTRODUCTION

Studies of the physical and chemical properties of silica surfaces have been carried out for nearly two centuries because it is the most widespread mineral on the Earth. The properties of silica depend on its forms which can be crystalline, as in crystalline silica particles, or amorphous, as in fused silica. Its surface can be hydrophobic when the surface chemical groups are mainly siloxane (Si-O-Si) groups, or hydrophilic

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when the surface possesses mainly silanol (Si-O-H) groups (Iler 1979). Therefore, according to conditions silica can reveal different surface properties which influence interparticle interactions in the dispersion system. The interactions are considered as attractive van der Waals and repulsive forces due to a layer of structured water at silica-water interface (Churaev 1999) and (Elimelech 1990). Different studies and various techniques are used to evidence these forces which can include measurements of colloidal stability (Elimelech 1990) and (Allen et al. 1970) and force measurements (Churaev 1999), (Elimelech 1990) and (Vigil et al. 1994). These forces play an important role in solid particle-liquid-solid particle interactions which influence numerous industrial processes.

The purpose of this paper is to determine the interaction forces between silica particles through liquid phase by measurements of the destruction time of the sediment silica column structure. Long chain alkanes were used as the liquid phase, which are often used as bridging liquid in all kinds of aggregation processes (Attita 1992), (Laskowski 1992), (Warren 1992) and (Sadowski 1994). The interactions among silica particles through the apolar phase will be discussed in this work.

## EXPERIMENTAL

### METHOD

A device for measurements of the destruction time of the sediment column structure was constructed in our laboratory (Waksmundzki et al. 1965) and (Ogonowski et al. 2001), and its schematic diagram is presented in Fig.1. The main part of the device is a glass tube (4) 0.4 m long and  $3 \times 10^{-3}$  m in inner diameter and a glass vessel (2)  $4.5 \times 10^{-5}$  m<sup>3</sup> in volume. The glass vessel is connected with the glass tube by a rubber plug (3) so as to move the tube up and down, and the vessel is closed by a stopper (1). The other end of the tube is closed by a silicone plastics seal (5) tightened by a screw (6).

### MEASUREMENTS

The destruction time of the sediment column structure was measured for high silica purity (>99.99%) which came from naturally occurring Russian silica particles (Caucasus). The silica powder, kindly supplied from the Glassworks "Ożarów" was fractionated by screening into four narrow particle size-fractions whose average diameters were:  $1.04 \times 10^{-4}$  m,  $1.35 \times 10^{-4}$  m,  $1.75 \times 10^{-4}$  m and  $2.25 \times 10^{-4}$  m, respectively. To remove contaminations of the particles surface occurring during screening, each fraction was treated by hydrochloric acid solution (1:1) for 24 h and next the particles were washed by redistilled and deionized water till its pH and conductivity were natural. The washed silica particles were heated for 5 h at 400° C and the dried products were stored in hermetically closed jars. The measurements of the destruction time of the sediment silica column structure for the studied size-

fractions were carried out in long chain alkanes from decane to hexadecane. Directly before the measurement each alkane used was purified by distillation and dried by a wide-porous silica gel.

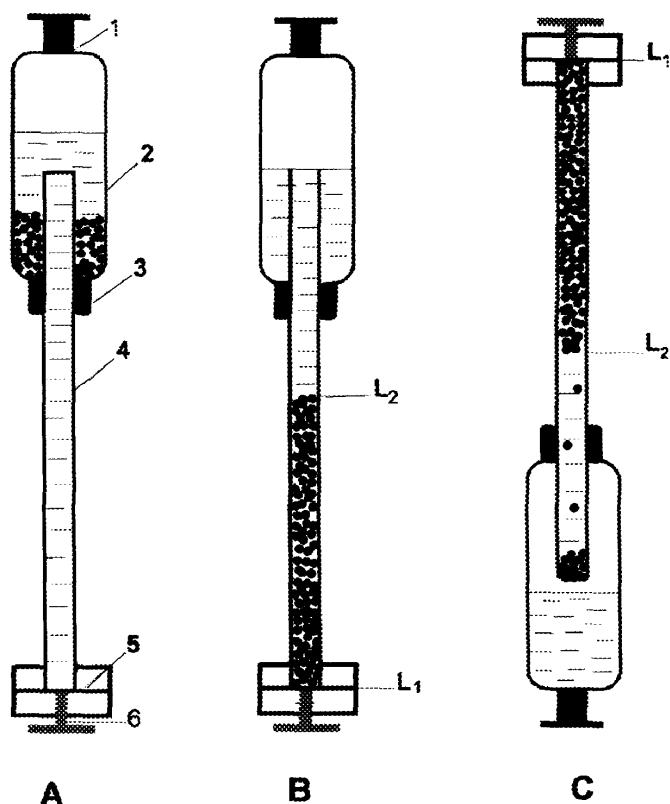


Fig. 1. A scheme of the device for destruction time measurements: (1) stopper, (2) glass vessel, (3) rubber plug, (4) glass tube, (5) silicone plastics seal and (6) screw. (A) the device, (B) formation of the sediment column, (C) destruction sediment column structure, L, and L<sub>2</sub> levels of the column sediments

The sediment column of silica particles was formed in the glass tube pouring  $3.0 \times 10^{-5} \text{ m}^3$  of the liquid in the glass vessel and adding  $1.388 \times 10^{-3} \text{ kg}$  sample of a given fraction of silica particles. The particles falling into the tube formed a sediment column and then the tube was moved up over the level of alkane (Fig. 1B), and the column structure was established in 10 min. Next, the device was quickly inverted as shown in Fig. 1C (the lower end of the tube must be over the liquid level) and the silica particles detached from the sediment column and dropped to the end of the tube, forming a new sediment column. The destruction time of the silica sediment column structure was measured by a stop-watch, starting when the first silica particle detached from the column at the level L<sub>2</sub> (Fig. 1C), and stopping when the last silica particle

started to drop at the level  $L_1$  (Fig. 1 C). The measurements of the destruction time were repeated several times for each silica fraction and each liquid used. The reproducibility of the destruction time measurements was very good.

## RESULTS

The measured values of the destruction time of the silica sediment column structure were expressed for the sediment column formed of  $1 \times 10^{-3}$  kg silica sample. The obtained results are presented as a function of the bulk properties of the components of the studied system, i.e. viscosity and density of alkanes and average diameter of the fractions of silica particles. The destruction time ( $t$ ) of the silica column structure and reciprocal of the destruction time ( $1/t$ ) as a functions of the viscosity and density of the alkanes are presented in Fig. 2 and 3, respectively. Curves 1,2,3 and 4 show the changes of the destruction time and the reciprocal of the destruction time of the sediment column structure of silica particles having average diameters of  $1.04 \times 10^{-4}$  m,  $1.35 \times 10^{-4}$  m,  $1.75 \times 10^{-4}$  m and  $2.25 \times 10^{-4}$  m, respectively. As can be seen the destruction time increases linearly with increasing viscosity of alkanes (Fig. 2). A similar relationship of the destruction time as a function of density is non linear and it is not presented here, but it appeared that the reciprocal of the destruction time ( $1/t$ ) as a function of the density is linear (Fig. 3). From Fig. 2 and 3 it can be seen that the destruction time decreases with increasing size of silica particles.

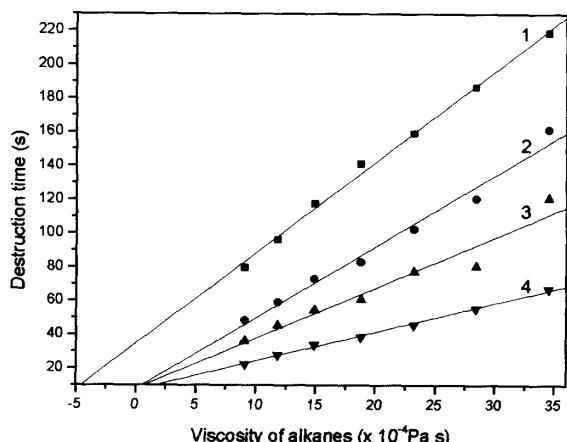


Fig. 2. Relationship between the destruction time ( $t$  in s) of the silica sediment column structure and viscosity of alkanes (from decane to hexadecane). Curves 1, 2, 3 and 4 represent the changes in  $t$  obtained for silica particles having the following average diameters:  $1.04 \times 10^{-4}$  m,  $1.35 \times 10^{-4}$  m,  $1.75 \times 10^{-4}$  m and  $2.25 \times 10^{-4}$  m, respectively.

## DISCUSSION

The fact suggests that the difference between detachment and attachment forces of silica particles through the alkane increased with its increasing viscosity and density, and decreased with increasing average diameter of silica particles (Fig. 2 and 3). So, for some diameters of silica particles the destruction time is infinitely long, and the particles do not detach from those in the column.

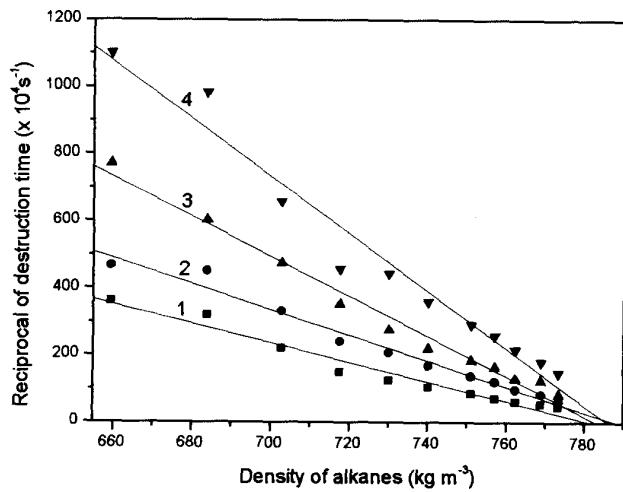


Fig. 3. Reciprocal of the destruction time ( $1/t_1$  in  $1/s$ ) as a function of the density of alkanes. Curves 1, 2, 3 and 4 represent the changes in  $1/t$  obtained for silica particles having the following average diameters:  $1.04 \times 10^{-4}$  m,  $1.35 \times 10^{-4}$  m,  $1.75 \times 10^{-4}$  m and  $2.25 \times 10^{-4}$  m, respectively

The diameter of such particles is called the critical diameter ( $d_{cr}$ ) (Waksmundzki et al. 1965) and (Waksmundzki 1965a) whose value can be determined from linear relationship between the reciprocal of the destruction time of the sediment column structure and average diameter of silica particles. The relationship is presented in Fig. 4. Curves from 1 to 7 were obtained for alkanes from decane to hexadecane, respectively, and they satisfy the linear equation

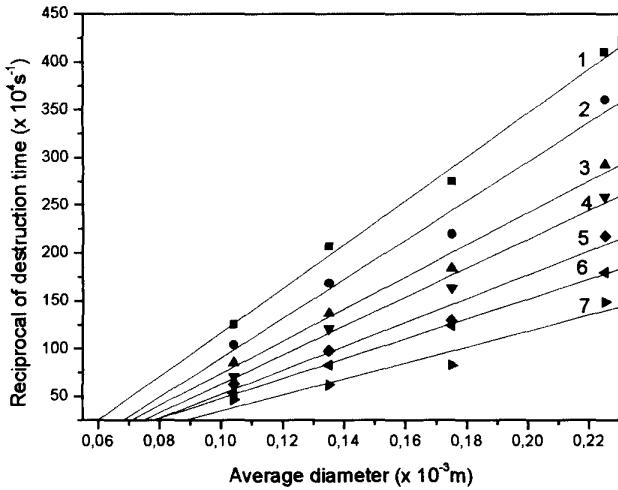


Fig. 4. Reciprocal of the destruction time ( $1/t$  in  $1/s$ ) as a function of the average diameter ( $d$  in m) of the silica particles. The  $t$  values were measured in decane (curve 1) undecane (2), dodecane (3), tridecane (4), tetradecane (5), pentadecane (6) and heksadecane (7)

$$\frac{1}{t} = a + bx \quad (1)$$

where  $t$  is the destruction time,  $a$  and  $b$  are the constants,  $x$  is the independent variable and in this case it is the average diameter ( $d$ ) of silica particles.

The values of the critical diameter of silica particles calculated from Eq. 1 are listed in Tab. 1. As we can see, the values of the critical diameter do not depend on the number of carbon atoms in the molecule of the studied alkanes, and the average value is  $5.6 \times 10^{-5}$  m. The silica particles having a diameter  $\leq d_{cr}$  do not detach from those of the column.

For silica particles having the critical diameter the attachment force of particles through the alkane should be equal to the detachment force, and they do not detach from those in the column. The detachment force of silica particles from those forming the sediment column in the alkane can be calculated from the following equation (Wójcik et al. 2000)

$$F_D = mg - V\rho_H g = \frac{4}{3}\pi d_{cr}^3 (\rho_S - \rho_H) \quad (2)$$

where  $m$  is the silica particle mass,  $g$  is the acceleration due the gravity,  $V$  is the silica particle volume,  $\rho_S$  and  $\rho_H$  are the density of silica and hydrocarbon, respectively, and  $d_{cr}$  is the average critical diameter.

Assuming that the silica particles are spherical for which  $d_{cr} = 5.6 \times 10^{-5}$  m and  $\rho_S = 2476 \text{ kg/m}^3$  (Poradnik, 1974) the detachment forces ( $F_D$ ) were calculated from Eq. 2. The values of  $F_D$  are listed in Tab. 1. and as we can see the detachment forces per particle decreased from  $17.28 \times 10^{-10}$  N to  $16.89 \times 10^{-10}$  N.

The  $F_D$  values (Tab. I) for the critical diameter of the silica particles depend on the density of the studied alkanes. The relationship between the reciprocal of the destruction time ( $\frac{1}{t}$ ) and the density of alkanes ( $\rho$ ) taken from literature (Janz et al. 1972) and (Wójcik et al. 2000), as it seen in Fig. 3, is linear, and the curves satisfy Eq. 1 in which the independent variable ( $x$ ) is the density of alkanes ( $\rho$ ).

In this case, like in the case of the average diameter of particle fractions, for an alkane in which the sediment column structure is stable, i.e. the silica particles do not detach from those in the column, its density is called critical ( $\rho_{cr}$ ). The values of the critical density calculated from Eq. 1 are listed in Tab. 2 and, as can be seen, they do not depend on the average diameter of the fractions of silica particles. The average value of the critical density of alkane is  $795.5 \text{ kg/m}^3$  (Tab. 2). It means that the sediment column of silica particles formed in the alkane of  $795.5 \text{ kg/m}^3$  density is stable. For such alkane the detachment force calculated from Eq. 2 is  $16.69 \times 10^{-10}$  N/particle, whose value as it is seen from Tab. 1, is minimal.

The minimal value ( $F_{DM}$ ) satisfies the equation (Wójcik et al. 2001) and (Ogonowski et al. 2001)

$$F_{DM} = W_{Coh,cr} \cdot L = W_{Coh,cr} \cdot 2\pi R \quad (3)$$

where  $F_{DM}$  is the minimal detachment force in alkane whose density and cohesion work of ( $W_{coh,cr}$ ) are critical,  $L$  is the perimeter of the contact plane between two silica particles in the alkane,  $R$  is the radius of the contact plane.

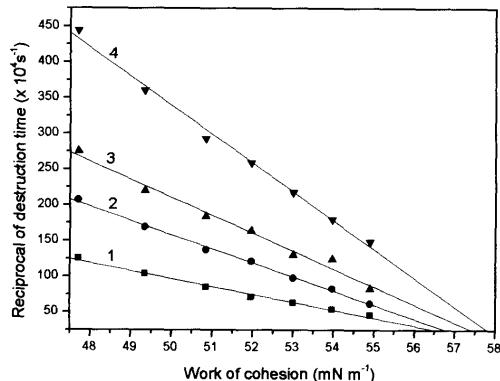


Fig. 5. Reciprocal of the destruction time ( $1/t$  in  $1/\text{s}$ ) as a function of the work of cohesion ( $W_{Coh}$ , in  $\text{mN/m}$ ). Curves 1, 2, 3 and 4 represent the changes in  $1/t$  obtained for silica particles having the following average diameters:  $1.04 \times 10^{-4} \text{ m}$ ,  $1.35 \times 10^{-4} \text{ m}$ ,  $1.75 \times 10^{-4} \text{ m}$  and  $2.25 \times 10^{-4} \text{ m}$ , respectively

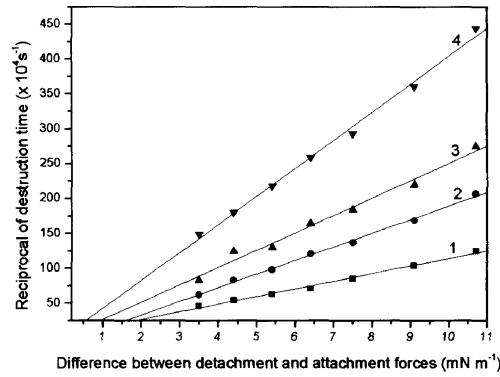


Fig. 6. Reciprocal of the destruction time ( $1/t$  in  $1/\text{s}$ ) as a function of the difference between the detachment and attachment force ( $\Delta F$  in  $\text{mN/m}$ ). Curves 1, 2, 3 and 4 represent the changes in  $1/t$  obtained for silica particles having the following average diameters:  $1.04 \times 10^{-4} \text{ m}$ ,  $1.35 \times 10^{-4} \text{ m}$ ,  $1.75 \times 10^{-4} \text{ m}$  and  $2.25 \times 10^{-4} \text{ m}$ , respectively.

To find the radius of the contact plane between two silica particles in n-alkane, the critical work of cohesion was determined from Fig. 5, which presents the relationship of the reciprocal of the destruction time of the silica sediment column structure as a function of the work of cohesion of alkanes (Wójcik et al. 2001). The curves in Fig. 5 are linear and they satisfy Eq. 1 in which the independent variable ( $x$ ) is the cohesion work of alkanes ( $W_{Coh}$ ). For the studied fractions the values of the critical cohesion work of the alkane calculated from Eq. 1 are listed in Tab. II and, as it is seen, they range from 58.0 to 58.8  $\text{mN/m}$ , and the average value is 58.4  $\text{mN/m}$ . Using this value and the detachment force from Tab. I ( $F_D$ ), the radii of the contact planes ( $R$ ) between two silica particles in the studied alkanes were calculated and they are listed in Tab I. The radii of the contact planes decrease from  $47.1 \cdot 10^{-10} \text{ m}$  to  $46.05 \cdot 10^{-10} \text{ m}$  with increasing work of alkane cohesion from 47.7 to 54.9  $\text{mN/m}$  (Tab.I). For the alkane having the critical cohesion work of 58.4  $\text{mN/m}$  the radius of the contact plane is  $45.5 \cdot 10^{-10} \text{ m}$ .

In the equilibrium conditions the attachment force between two silica particles in n-alkane is expressed in the form:

$$F_A = W_{Coh} \cdot 2\pi R \quad (4)$$

where  $F_A$  is the attachment force,  $W_{Coh}$  is the cohesion work of alkanes.

Table 1. Values of the critical diameter of silica particles ( $d_{cr}$ ) the detachment ( $F_D$ ) and attachment ( $F_A$ ) force, the radii ( $R$ ) of the contact plane and the difference ( $\Delta F$ ) between the detachment and attachment force, values of the  $F_D$ ,  $F_A$ ,  $R$  and  $\Delta F$  obtained for the critical parameters

Alkane	$d_{cr} \times 10^{-5} \text{m}$	$F_D \times 10^{-10} \text{N/particle}$	$R \times 10^{-10} \text{m}$	$F_A \times 10^{-10} \text{N/particle}$	$\Delta F \text{ mN/m}$
Decane	5.4	17.28	47.12	14.11	10.7
Undecane	5.6	17.19	46.87	14.52	9.1
Dodecane	5.6	17.09	46.60	14.88	7.5
Tridecane	5.8	17.04	46.46	15.16	6.4
Tetradecane	5.8	16.99	46.32	15.42	5.4
Pentadecane	5.4	16.93	46.16	15.64	4.4
Hexadecane	5.8	16.89	46.05	15.88	3.5
Average	5.6	16.69	45.5	16.69	0

Table 2. Values of average diameters of particle size-fraction ( $d_a$ ), and values of the critical: density ( $\rho_{cr}$ ), the work of cohesion ( $W_{coh,cr}$ ), the difference ( $\Delta F_{cr}$ ) between detachment and attachment forces

$d_a \times 10^{-4} \text{m}$	$\rho_{cr} \text{ kg/m}^3$	$W_{coh,cr} \text{ in mN/m}$	$\Delta F_{cr} \text{ mN/m}$
1.04	797.7	58.8	-0.43
1.35	793.3	58.0	0.31
1.75	795.6	58.4	-0.05
2.25	795.5	58.4	-0.05
Average	795.5	58.4	-0.055

Taking into account the radii of the contact planes from Tab. 2 and the cohesion work of alkanes from the literature (CRC Handbook 1972) and (Wójcik et al. 2000), the attachment forces were calculated from Eq. 4 and they are listed in Tab. 1. As can be seen the attachment force between two silica particles, in contrast to the detachment force, increases from  $14.1 \times 10^{-10}$  to  $16.7 \times 10^{-10} \text{ N/particle}$  with increasing cohesion work of alkanes.

The detachment and attachment forces were expressed in term of mN/m, and the differences between them ( $\Delta F = F_D - F_A$ ) were calculated and listed in Tab. 1. The relationship of the reciprocal of the destruction time as a function of the  $\Delta F$  is presented in Fig. 6. As it is seen the curves in Fig 6 are linear and they satisfy Eq. 1 in which the independent variable ( $x$ ) is the difference between the detachment and attachment force ( $\Delta F$ ). The critical values of  $\Delta F$  (the destruction time is equal to infinity) calculated from Eq. 1 are listed in Tab. 2, and, as we can see, their values do not depend on the average diameters of size-fractions of silica particles, and the

average value is small but negative. It may mean that detachment of silica particles from those in the sediment column occurred by disruption of the alkane film present between silica particles. Small negative values of  $\Delta F_{cr}$  suggest that the free energy of the alkane film is a little changed by the free energy of silica surfaces. Such small changes of the free energy of alkane film caused by the surface free energy of silica may result from a small difference between the surface free energy of alkane being the Lifshitz van der Waals energy and the Lifshitz van der Waals component of the surface free energy of silica.

## CONCLUSION

The presented analysis and discussion of the results of the detachment experiments has clearly shown that detachment of coal particle from one another in the studied liquids occurs by disruption of the liquid film present between them. The free energy of the alkane film is a little increased by the free surface energy of silica particles.

The perimeter of the contact plane between two coal particles decreases with increasing length of the hydrocarbon chain of alkanes and alcohols. Increase of the destruction time of the sediment column structure of coal particles with increasing length of the hydrocarbon chain of the studied liquids results on the one hand from decrease of the detachment force of coal particle and from one another and on other from an increase of the attachment force between them.

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**W. Wójcik, B. Jańczuk, R. Ogonowski, Oddziaływanie między ziarnami krzemionki w alkalicznym środowisku, Fizykochemiczne Problemy Mineralurgii, 36, 2002, 279-288 (w jęz. ang.)**

W pracy przedstawiono wyniki pomiarów czasu niszczenia struktury słupka sedymentu ziaren krzemionki w węglowodorach alifatycznych od dekanu do heksadekanu. Pomiary te przeprowadzono w tzw. „urce Waksmundzkiego” dla frakcji ziarnowych krzemionki, których średnie średnice wynoszą odpowiednio:  $1,04 \times 10^{-4}$  m,  $1,35 \times 10^{-4}$  m,  $1,75 \times 10^{-4}$  m i  $2,25 \times 10^{-4}$  m.

Uzyskane rezultaty czasu niszczenia struktury słupka sedymentu ziaren krzemionki omówione zostały w zależności od objętościowych właściwości krzemionki i alkanów, jak również w zależności od międzymolekularnych właściwości. Czas niszczenia struktury słupka sedymentu rośnie liniowo tylko ze wzrostem lepkości badanych alkanów, natomiast liniowe zmiany stwierdzone zostały dla zależności odwrotności czasu niszczenia struktury w funkcji średniej średnicy ziaren krzemionki, gęstości i pracy kohezji węglowodorów. Z tych liniowych zależności wyznaczona została krytyczna średnica ziaren, krytyczna gęstość i praca kohezji. W układach, w których jeden z parametrów ma krytyczną wartość, struktura słupka sedymentu jest trwała tzn. niemożliwe jest jej zniszczenie. Wówczas siły odrywu są równe siłom zlepienia. Zakładając, że ziarna krzemionki są kulkami obliczono siły odrywu, które nieznacznie maleją ze wzrostem długości łańcucha węglowodorowego alkanów. Wykorzystując wyznaczoną krytyczną wartość pracy kohezji, która praktycznie nie zależy od wielkości ziaren krzemionki, obliczono promień płaszczyzny kontaktu pomiędzy ziarnami krzemionki. Znając wielkość promienia płaszczyzny kontaktu obliczono siły zlepienia pomiędzy ziarnami krzemionki w alkanach.

Siły zlepienia, w przeciwieństwie do siły odrywu, rosną ze wzrostem długości łańcucha węglowodorowego alkanów, w rezultacie różnica pomiędzy nimi maleje od  $3,2 \times 10^{-10}$  N/ziarno (10.7 mN/m) dla dekanu do zera dla węglowodoru o krytycznej pracy kohezji.

Na podstawie liniowych zmian odwrotności czasu niszczenia struktury słupka sedymentu krzemionki w funkcji różnicy sił odrywu i zlepienia wyznaczono krytyczną wartość tej różnicy. Niewielkie ujemne wartości krytycznej średnicy świadczą, że energia filmu węglowodorowego pomiędzy ziarnami krzemionki jest nieco zwiększoną przez swobodną energię powierzchniową krzemionki.