

Waksmundzki's tube – a device for studying a dispersion system

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The goal of the paper is to review the results of the destruction time of the solid sediment column structure measurements using a simple device which was elaborated by Waksmundzki and co-workers. The authors and Russian researchers used the device to determine the influence of the flotation reagents on the stability of the dispersion system and the obtained results are shortly described. Next, we summarized the results of the destruction time of coal and silica sediment column structures formed in two homologous series of liquids i.e. alkane and alcohol. On the basis of the results the manner of the calculation of the detachment and attachment forces in the system solid particle/liquid (alkane or alcohol) /solid particles is also presented. Some unpublished results were advantageous to explanation of the changes of the attachment and detachment forces.

1. INTRODUCTION

Adhesion of small particles to one another or a solid surface through a liquid film and disruption of their contact is of a great scientific and technological interest. For example, the efficiency of solid-liquid separation (agglomeration, flocculation, coagulation, sedimentation, filtration, flotation etc.) depends, among other factors, on particle/liquid film/particle interactions [1-6]. The interactions are difficult to characterize; therefore different theoretical models and very complicated techniques and expensive apparatuses were used for studies [7-16].

Theoretical models for the adhesion force between particles date back over 70 years. The models were the investigation object of Hertz [11], Bradley [12], Derjaguin et al. [13,14] and Johnson et al. [15], which were then intensively

developed by many researchers [16-21]. To determine the magnitude of the adhesion forces between particles and various substrates, different techniques and instruments such as surface force apparatus [22,23], atomic force microscopy [7,10], scanning electron microscopy [7,24], hydrodynamic methods [25,26], rotating disk surface [27] and others [13,14,26] were used. Waksmundzki et al. [28,29] elaborated a very simple and inexpensive device to study the stability of the dispersion systems by measuring the destruction time of a sediment column structure.

A device for destruction time measurements of the sediment column structure. Among Polish researchers a device is known as “Waksmundzki’s tube”. The aim of this paper is to summarize the results of the destruction time of the sediment column structure measurements using the device described in [28-45].

A schematic diagram of this device is presented in Figure 1. It consists of a glass tube (4) 0.4 m long the inner diameter of which can range from 2.6×10^{-3} m to 3.2×10^{-3} m, and a glass vessel (2) which volume can range from 3×10^{-5} m³ to 4.5×10^{-5} m³. The glass vessel is connected with the glass tube by a rubber plug (3) so as to move the tube up and down. The glass vessel is closed by a stopper (1) and the other end of the tube is closed by a silicone plastic seal (5) tightened by a screw (6).

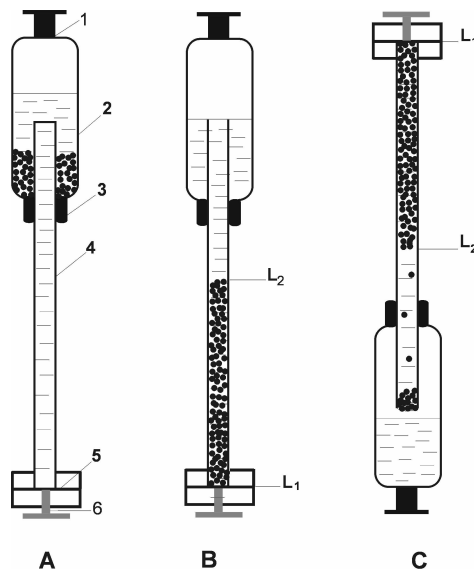


Fig. 1. A scheme of the “Waksmundzki’s tube” for destruction time measurement: (1) stopper, (2) glass vessel, (3) rubber plug, (4) glass tube, (5) silicone plastics seal, and (6) screw (from Ref. 42) .

Destruction time measurement. The experiments of the destruction time of the sediment column structure are carried out for various size-fractions of mineral particles [28,29]. The sediment column of the particles is formed by pouring ca $2.5 \times 10^{-3} \text{ m}^3 - 3.0 \times 10^{-3} \text{ m}^3$ of a liquid in the glass vessel and a tube and then adding strictly weighed sample of a given fraction of the particles (Fig. 1A). Next, the glass tube moves down as shown Figure 1B, and the particles falling into the tube form a sediment column. Then the tube moves up over the level of the liquid and the column structure is established at a strictly definite time. After that the device inverts quickly as shown in Figure 1C, (the lower end of the tube must be over the liquid level) and single particles detach from those in the column and drop to the end of the tube, forming a new sediment column. The destruction time of the sediment column structure is measured by a stop-watch, starting when the first particle detaches from the column (Fig. 1C), and stopping when the last particle starts to drop.

Measurement conditions. At first, Waksmundzki et al. [28,29] determined the optimal measurement conditions of the destruction time of the sediment column structure of silica particles in water. Experiments were carried out using different glass tubes and different size-fractions of silica particles. The authors of the device found that for all studied size-fractions and glass tubes the column structure equilibrium was established during a few minutes, and this time was determined for 10 minutes [28,29]. Moreover, the destruction time of the silica column structure depends on the inner diameter of the used glass tubes, but it does not depend on their length. The destruction time of the silica column structure in water increased with decreasing inner diameter of the glass tubes and the size-fractions of silica particles. In a small range of silica particles the destruction time increases asymptotically to the terminal value, so for the smallest ones their value becomes infinitely long. To determine this value Waksmundzki et al. [28,29] took advantage of the linear relationship between the reciprocal of the destruction time and diameter of silica particles. This relationship [28] is presented in Figure 2 for the results obtained in glass tubes of different inner diameters.

The curves in this figure satisfy the equation

$$\frac{1}{t} = a + b \cdot x \quad (1)$$

where t -is the destruction time, a and b are the constants and x is the diameter of silica particles (independent variable).

As it is seen from Figure 2, for the destruction time equal infinity ($1/t = 0$) silica particles did not detach from those of the sediment column, and the

diameter of such particles was called critical. The critical diameter of silica particles, calculated from Eq. 1, did not depend on the inner diameter of the used glass tube, and the value was equal 5.3×10^{-5} m [28,29]. It means that for determination of the critical diameter of solid particles we only need to measure the destruction time in one tube. The structure of the sediment column formed from silica particles smaller than or equal the critical diameter is stable. In this case the attachment force between two silica particles through water film is higher than or equal the detachment force. In further studies the authors [30] determined the critical diameter for particles of a few minerals (barite, fluorite and galena), and next they found that the collectors usually used to enrich these minerals markedly changed the critical diameter. Moreover, the relationship between the destruction time and concentration of the collector is a polyextremal function. The fact indicates that at some concentrations of the collector flocculation or deflocculation occurs.

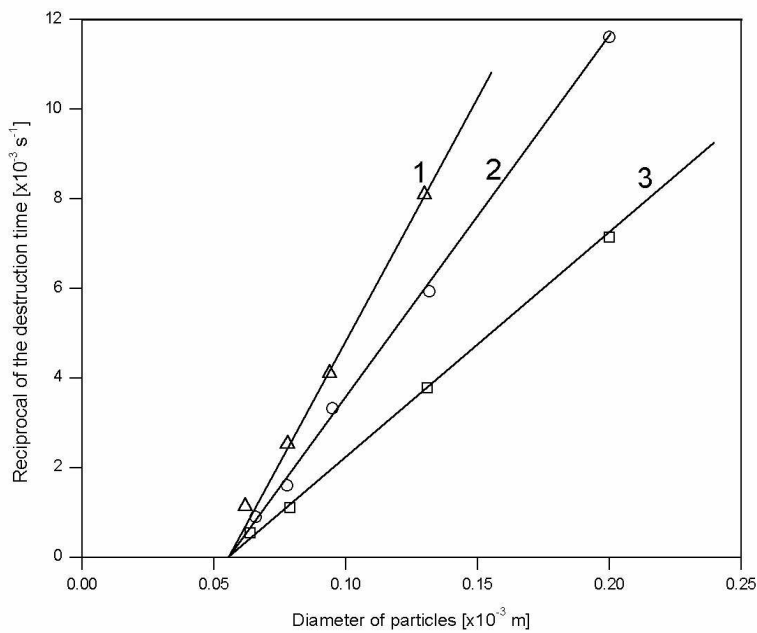


Fig. 2. Reciprocal of the destruction time ($1/t$ in $1/s$) as a function of the average diameter (d in m) of silica particles. The t values were measured in the glass tube having the inner diameters: 3.2×10^{-3} m (curve 1), 2.8×10^{-3} m (2) and 2.3×10^{-3} m (3) (from Ref. 29).

Using the device by others. A few years later Russian researchers [31] modified “the Waksmundzki tube” for studying the stability of mineral water suspensions. They [31] changed the procedure of the destruction time measurement.

According to them the device was filled with a liquid, so during measurements the particles detached from those in the sediment column fell to the bottom of the glass vessel without forming another sediment column. Such a measurement modification does not practically change the values of the measured destruction time of the sediment column structure.

Klassen et al. [32,33] used “the Waksmundzki tube” to study the influence of: 1) the concentration of different flocculation depressors on the stability of magnetite suspension, 2) the intensity of the magnetic field on the stability of water apatite suspension. They found that deflocculation properties of the studied depressors were revealed at their low concentrations, and above some concentrations the properties decreased. The water glass appeared to be the best deflocculation reagent among the studied depressors. Pretreatment of apatite suspensions in the magnetic field caused increase of the critical radius of apatite particles from 20 μm to 35 μm . Moreover, the influence of the intensity of the magnetic field on the stability of apatite suspension is bigger for smaller particles. So, for particles smaller than 150 μm the destruction time is ca. 20-25% higher than for particles which diameter is bigger than 150 μm . According to the opinion of Klassen et al. [32,33] explanation of the above mentioned facts were the changes of hydration of apatite surface occurring under the influence of the magnetic field intensity.

Influence of a liquid film on stability of the dispersion system. Different reagents (collector, frother, modifier, depressor) are used for separation of a valuable component from ore-bearing rock by flotation or agglomeration processes [1-6]. Alkanes and alcohols were commonly employed as collector models (or bridging liquid) or frother for beneficiation of hydrophobic or hydrophobized minerals.

Therefore, it was interesting from the theoretical and practical point of view to determine the value of the adhesion force between mineral particles through a bridging liquid film. To solve this problem we again used [34,35] “the Waksmundzki tube” as a very simple and cheap method. The destruction time of the sediment column structure formed of coal particles pre-covered with an alkane or alcohol film of various thickness was measured first [34,35]. It appeared that thickness increase of both alkane or alcohol film caused the destruction time increase, but the critical diameter of coal particles did not depend on the thickness of the film. On the basis of the obtained results, their analyses and discussion we found that detachment of coal particles from those in the column occurred via disruption of the film (alkane or alcohol) present between them, and the changes of the detachment and attachment force depended on the contact plane between two coal particles which value depended

on the length of hydrocarbon chains of the alkane or alcohol [34,35]. Unfortunately, we were not able to calculate the value of contact plane because the studied systems were too complicated.

Interparticle action in different liquid media. To confirm the conclusion concerning the disruption of the liquid film and the contact plane size between two particles, detachment experiments were carried out for systems involving two kinds of solid: coal as hydrophobic, and silica as hydrophilic example in two homologous liquid series, i.e. alkanes, from hexane to hexadecane, and alcohols, from ethanol to decanol [36-45]. The experimental procedure of the destruction time measurement, the obtained results and their discussion is described in papers [36-45]. From the results we found that the destruction time of the sediment column structure of both solids increased with increasing number of carbon atoms in the molecule of the studied liquids and with decreasing diameter of the solid particles. As example, the relationship between the destruction time of the silica sediment column structure and the average diameter of the fractions of the silica particles in Figure 3.

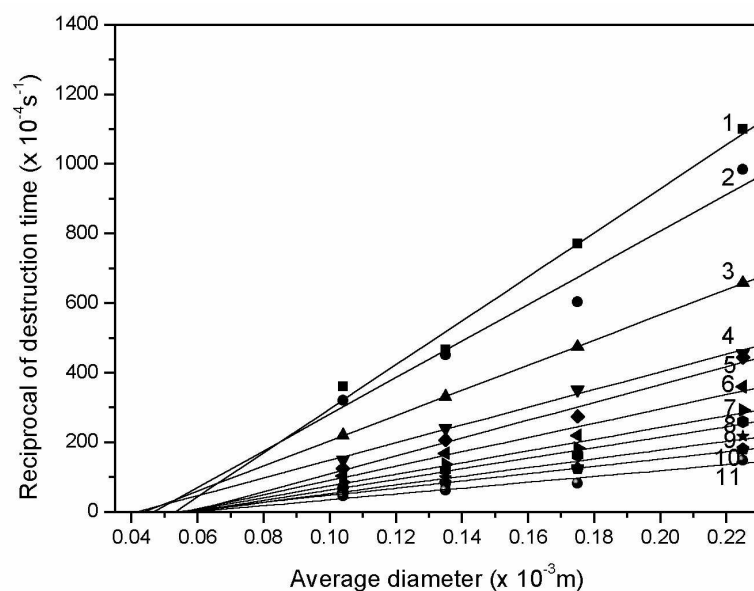


Fig. 3. Reciprocal of the destruction time ($1/t$ in $1/s$) as a function of the average diameter (d in m) of silica particles. The t values were measured in hexane (curve 1), heptane (2), octane (3), nonane (4), decane (5), undecane (6), dodecane (7), tridecane (8), tetradecane (9), pentadecane (10) and hexadecane (11) (from Ref. 43).

We found that, apart from the earlier stated linear relationships between the reciprocal of the destruction time and the average diameter of the particles fractions [28-30], the same linear dependence existed between $1/t$ and work of cohesion of the liquids and their density and the free energy interaction [40-45]. Therefore, from Eq. 1 not only the critical diameter but also the critical density, work of cohesion and free energy interaction were calculated.

It appeared that the calculated critical diameter, critical work of cohesion and critical density for both homologous series of liquids and solids did not depend on the length of the hydrocarbon chain of the liquid and on the diameter of the particle fractions, respectively. Therefore, the average values of the critical parameters, listed in Table 1, were used in the discussion of the dispersion system stability [36-45]. As it is seen, the critical diameter of coal particles is nearly two times bigger than that for silica particles. In the case of coal the average critical diameter in alkanes is lower than that in alcohols, but for silica particles the dependence is reverse.

Tab. 1. Average values of: diameter, work of cohesion and density obtained for coal and silica in alkanes (A) and alcohols (B).

No.	Critical parameter	Coal		Silica	
		A	B	A	B
1	Diameter in $m \times 10^{-5}$	9.4	9.8	5.3	5.0
2	Work of cohesion in mJ/m^2	57.3	58.4	56.0	59.4
3	Density in kg/m^3	789.1	835.9	784.8	834.9

Such sequence for the critical work of cohesion and the critical density does not hold, and all the critical values in alcohols are bigger than those in alkanes. It should be emphasised that the critical diameter of coal and silica in water is $10.0 \times 10^{-5} m$ and $5.9 \times 10^{-5} m$ [38,41,45], respectively. The particles having a diameter \leq than critical ones do not detach from those in the column. For such particles the attachment force between them through liquid (alkane or alcohol) should be equal to the detachment force. The sediment column formed in liquid having the critical work of cohesion and density the particles do not also detach from one another. On the basis of the relationships between the work of cohesion of alkanes and number of carbon atom in molecule of alkanes the coal and silica particles do not detach from those in the column if the column is formed in eicosane and octadecane, respectively. This result is unexpected because the density of silica is nearly two times bigger than coal. In the case of alcohols the stable structure of the sediment column is in tridecanol.

The above mentioned facts and the values of the critical parameters clearly indicate that the stability of the studied dispersion systems depends not only on

the inertial forces but also on the interfacial forces. It also results from the differences between the density of the studied solids (coal and silica) and liquids (alkanes, alcohol), that would suggest proportionality of the differences and the values of the average critical diameter of coal and silica particles.

Determination of the detachment and attachment forces. The destruction time of the sediment column structure depends on the magnitude of the attachment and detachment force. Assuming that the solid particles are spherical the detachment force can be calculated from the equation:

$$F_D = mg - V\rho_L g = \frac{1}{6}\Pi d^3 g(\rho_S - \rho_L) \quad (2)$$

where m is the solid particle mass, g is the acceleration due the gravity, V is the solid particle volume, ρ_S and ρ_L are the density of solid and liquid, respectively, and d is the particle diameter.

For the solid particles having the diameter equal to the critical diameter the detachment force is equal to the attachment force, and they satisfy the equation:

$$F_D = F_A = W_{SL,Cr} \cdot L = W_{SL,Cr} \cdot 2\Pi R \quad (3)$$

where F_A is the attachment force, $W_{SL,Cr}$ is the critical work of cohesion of the liquid, L is the perimeter of the contact plane between two solid particles through the liquid film and R is the radius of the contact plane.

In equilibrium conditions the attachment force between two solid particles through a liquid film is expressed in the form:

$$F_A = W_{SL} \cdot 2\Pi R \quad (4)$$

where W_{SL} is the work of cohesion of liquid.

At first the detachment forces for all solid systems were calculated from Eq. 2 for solid particles which average diameter was critical. Next, using the critical work of cohesion, the radii of the planes were calculated from Eq. 3, and then from Eq. 4 the attachment forces were calculated.

Details of these calculations are described in the papers [36-45]. Here in Table 2 only the values of the bottom and top limits of the detachment and attachment force and their differences are presented

Tab. 2. Values of the detachment (F_D) and attachment (F_A) forces and the difference (ΔF) between F_D and F_A obtained for coal (A) and silica (B).

Lp	Liquid	Coal (N/particle) x 10 ⁻¹⁰			Silica (N/particle) x 10 ⁻¹⁰		
		F _D	F _A	ΔF	F _D	F _A	ΔF
1	Hexane	29,10	18,70	10,40	15,20	9,90	5,30
⋮	↓	↓	↓	↓	↓	↓	↓
2	Hexadecane	24,20	23,20	1,00	14,30	13,90	0,40
3	Extreme	23,50	23,50	0,00	14,20	14,20	0,00
4	Ethanol	26,70	20,40	6,20	12,00	9,05	2,95
⋮	↓	↓	↓	↓	↓	↓	↓
5	Decanol	24,7	24,00	0,70	11,74	11,19	0,55
6	Extreme	24,6	24,60	0,00	11,70	11,70	0,00

The detachment forces and the radii of the contact plane for both solid and homologous series of liquids decreased, whereas the attachment forces increased with increasing length of the hydrocarbons chain of the liquids. The increase of the destruction time of the sediment column structure of the studied solids particles with increase of the density and work of cohesion of the liquids results, on the one hand, from a decrease of the detachment force of solid particles from one another, and, on the other, from an increase of the attachment force between solid particles. As we can see from Table 2, for systems coal particles/liquid/coal particles having critical parameters the attachment forces among coal particles in alcohol are bigger than those in alkanes, but in systems involving silica particles the sequence is reverse. Moreover, the force values for systems involving coal are nearly two times bigger than those of the silica. The difference in the properties of the studied two systems is also exhibited by the relationship of the reciprocal of the destruction time as a function of the differences between the detachment and attachment forces. The values difference between the forces for both studied systems were small and negative with exception of the smallest fraction of silica whose value was small but positive. The negative values of these differences can evident that the free energy of the liquid films present between solid particles is slightly increased by the surface free energy of solids [36-44].

It is evident that for the above calculation interaction electrostatic was not taken into account. This suggestion is confirmed by the results of the measurements of the destruction time of the silica sediment column structure in NaCl and CaCl₂ solution, which have not been published yet. In this case the differences between the forces (F_D and F_A) for $1/t = 0$ were negative and big enough.

In these studies another problem appeared. To determine the radius of the contact plane the critical work of cohesion was calculated on the basis of linear dependences between the reciprocal of the destruction time and the work of cohesion of alkanes and alcohols. We found that the above mentioned relationship consists of two exact linear ranges. In consequence two values of the critical work of cohesion for alkanes and alcohols were obtained. The first was from hexane to nonane and from ethanol to pentanol for homologous series of alkane and alcohol, respectively. We shall now try to solve these two problems, but it more sophisticated experiments must be carried out.

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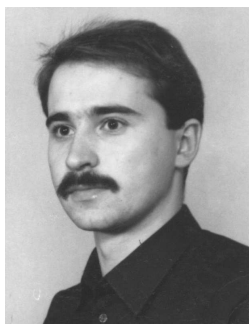
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