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The influence of Ca²⁺ and La³⁺ and magnetic field on the surface free energy components of alumina

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A novel method based on the imbibition and drainage of the probe liquid into a porous bed was applied to determine surface free energy components of alumina. Apolar Lifshitz-van der Waals component, and electron-donor and electron-acceptor parameters of polar (acid base) component were determined from weight of probe liquids remained in the porous bed of bare alumina. The influence of Ca²⁺ and La³⁺ ions and magnetic field effect on these components were investigated. Both, the presence of deposited ions and magnetic field, influence on changes in electron-donor and electron-acceptor parameters, which resulted from the presence of hydrogen bonding on the alumina surface. The observed changes in acid-base interactions (hydrogen bonding) result from changes in the structure of the water film at the surface.

1. INTRODUCTION

Surface free energy and its components are important parameters characterizing surface properties of solids and play a significant role in various interfacial phenomena such as adsorption, wetting, spreading and adhesion (in catalysis, aggregation, filtration, flotation or colloid stability). In recent years numerous attempts have been made to describe it quantitatively in various systems, using different approaches to express interfacial interactions [1–5]. Moreover, studies have been intensified, which concerned not only mathematical description of interfacial solid/gas and solid/liquid interactions, but also

experimental determination of solid surface free energy [6–12]. The estimation of its real values, however, is still an open problem, which results from the fact that indirect methods are used, which are mostly based on the Young equation and contact angle measurements [6,10,11]. It is necessary to obtain a flat solid surface for these methods, however is not always possible, e.g. for solids naturally occurring in the form of powder. Methods based on imbibition of probe liquids into a thin porous layer deposited on a glass plate (thin layer wicking method – TLWM [8]) or powdered solid packed in a glass capillary (thin column wicking method – TCWM [9]) are usually applied. The liquid penetration rate is described by Washburn equation [13].

Recently Chibowski et al. [10,14] proposed a novel method (imbibition—drainage method – IDM) for powdered solids for determination of the surface free energy components. The method is a modification of TCWM and based on imbibition of probe liquids into the investigated solids porous bed. Theoretical background of imbibition—drainage method is based on the theory of capillary rise and liquid drop detachment from a capillary [13]. Liquid entrees into a capillary due to the Laplace pressure present across the meniscus and the total weight *W* of the column of liquid in the capillary describes Eq. (1):

$$W = mg = 2\pi r \gamma_L \cos \theta \tag{1}$$

where: m – the mass of the liquid in the capillary, g – the acceleration of gravity, r – the capillary radius, χ – the liquid surface tension, θ – the contact angle between the tangent to the liquid meniscus and the capillary wall.

Then, in Eq. (1) $\cos \theta = 1$, e.g. the liquid completely wets the capillary walls, then it is read:

$$W = mg = 2\pi r \gamma_{I} \tag{2}$$

Eq. (2) is strictly fulfiled only if the liquid completely wets the surface and a precursor duplex film is present on the capillary wall.

If the liquid does not wet the surface completely, then it moves into the capillary (or porous bed) at dynamic advancing contact angle, θ_a and the total changes in free energy, ΔG_a (per unit surface) at the interface solid/liquid in the capillary take place [10]:

$$m_a g = 2\pi r \gamma_L \cos \theta_a = 2\pi r \Delta G_a \Rightarrow \gamma_L \cos \theta_a = \Delta G_a = \frac{m_a g}{2\pi r}$$
 (3)

The energy changes ΔG_r during the liquid drainage from the capillary at dynamic receading contact angle θ_r (or porous bed) can be expressed in a similar way:

$$m_r g = 2\pi r \gamma_L \cos \theta_r = 2\pi r \Delta G_r \Rightarrow \gamma_L \cos \theta_r = \Delta G_r = \frac{m_r g}{2\pi r}$$
 (4)

These specific free energy changes accompanying the capillary imbibition ΔG_a , and its drainage ΔG_r , can be used for the solid surface free energy determination:

$$\Delta G_{a} - \Delta G_{r} = W_{A} - W_{C} \quad \Rightarrow W_{A} = \Delta G_{a} - \Delta G_{r} + W_{C}$$
 (5)

where: W_A – the work of adhesion of the liquid and W_C – the work of cohesion.

Using the approach of van Oss *et al.* [4] the work of adhesion can be expressed by the surface free energy of the solid and the liquid:

$$W_{A} = 2 \left[\left(\gamma_{S}^{LW} \gamma_{L}^{LW} \right)^{1/2} + \left(\gamma_{S}^{+} \gamma_{L}^{-} \right)^{1/2} + \left(\gamma_{S}^{-} \gamma_{L}^{+} \right)^{1/2} \right]$$
 (6)

where: γ^{LW} is the apolar Lifshitz-van der Waals component of surface free energy, γ and γ^{+} are the electron-donor and electron-acceptor parameters of acid-base component of surface free energy, subscript s stands for solid and subscript l stands for liquid.

The purpose of this work was to apply the novel method based on imbibition of the solid porous bed and the filtration (drainage) of liquid for determination of the alumina surface free energy components whose surface was modified with Ca²⁺ and La³⁺ ions and exposed to a magnetic field of two S–S poles and 0.1 T magnetic induction.

2. EXPERIMENTAL DETAILS

Materials. To test the ID method alumina $(Al_2O_3\ 90\ aktiv\ basic, Merck; 0.063–0.2 mm) was used. Before it use to the experiment alumina powder was dried at 50°C for 0.5 h and then at 150°C for 1.5 h and stored in a desiccator at room temperature.$

Ion doping. The alumina samples of 6.4 g were placed into 40.2 cm^3 of 10^{-3} M solutions of $CaCl_2$ and $LaCl_2$ then water was evaporated at $80^{\circ}C$ for 0.5 h. The samples were then placed in an oven at $200^{\circ}C$ for 2 h in order to eliminate the residual water.

Methods. To determine the surface free energy components of alumina, measurements of the liquid weights in the porous bed for the advancing (A) and receding (R) modes were performed.

- Advancing mode (determination of m_a). 0.4 g samples of alumina were weighed out into plastic tips applied for automatic pipettes (blue, 2 ml from Aldrich). The outlet of each tip used was blocked with a tiny swab of Nylon fibers to avoid any leakage of the powder during the experiments. In order to achieve standardized filling a vibrator was used. The tip outlet was dipped 2–3 mm into the liquid in a vessel. After the bed had been wetted with the liquid, it was weighed in order to find amount of the liquid remained in the powder.
- **Receding mode** (determination of m_r). Several drops of the same liquid were dropped onto the bed top and weigh until the "filtration process" was finished. Then the tip was weighed again.

These experiments were repeated three to five times and the average values were taken for the calculations. The probe liquids used for experiments are listed in Table 1 [15].

Tab. 1. Surface tension of probe liquids, γ_L , and its components: Lifshitz-van der Waals, γ^{LW} , electron acceptor, γ^+_L , and electron-donor, γ^-_L , (mN/m).

Liquid	Origin and purity	$\gamma_{ m L}$	$\gamma^{ m LW}$	γ ⁺	γ
n-Nonane	Fluka, >99%	22.9	22.9	0	0
n-Decane	Merck-Schuchard, >99%	23.8	23.8	0	0
n-Dodecane	Fluka, >99%	25.35	25.35	0	0
Diiodomethane	POCh Gliwice (Poland), p.a.	50.8	50.8	0	0
Water	Mili-Q Plus system	72.8	21.8	25.5	25.5
Formamide	RCB (Belgium) p.a.	58.0	39.0	2.28	39.6

Magnetic field. Magnetic field originated from two S–S type permanent magnets, 0.1 T each, kindly supplied by the magnetizer Group Inc. USA. Effects of magnetic field on the components of surface free energy of alumina were determined by IDM for the systems:

- pure Al₂O₃,
- Al₂O₃ modified with Ca²⁺ (Al₂O₃/Ca²⁺),
- Al_2O_3 modified with La^{3+} (Al_2O_3/La^{3+}).

In this purpose the samples of alumina powder were placed between the poles of the magnetizer and the measurements of imbibition and drainage of probe liquids in the porous bed for the advancing and receding modes, respectively, were investigated.

All experiments were repeated 3–4 times and the average values together with the standard deviations were calculated.

Calculations. The effective radius, $R_{\rm ef}$, of alumina porous bed was determined from receding mode of n-alkanes:

$$R_{ef} = \frac{m_r g}{2\pi \gamma_L} \tag{6}$$

The apolar Lisfhitz-van der Waals component, γ^{LW}_{S} , was determined from advancing and receding modes of diiodomethane:

$$\Delta G_{a} - \Delta G_{r} = 2 \left(\gamma_{S}^{LW} \gamma_{L}^{LW} \right)^{1/2} - 2 \gamma_{L} \quad \Rightarrow \left(\gamma_{S}^{LW} \right)^{1/2} = \frac{\frac{g}{4\pi R_{ef}} (m_{a} - m_{r}) + \gamma_{L}}{\left(\gamma_{L}^{LW} \right)^{1/2}}$$
(7)

The electron-donor, γ_S , and electron-acceptor, γ_S^+ , parameters of acid-base component were obtained by simultaneous solving of two equations, from advancing and receding modes of water and formamide:

$$W_{A} = 2\left[\left(\gamma_{S}^{LW}\gamma_{L}^{LW}\right)^{1/2} + \left(\gamma_{S}^{+}\gamma_{L}^{-}\right)^{1/2} + \left(\gamma_{S}^{-}\gamma_{L}^{+}\right)^{1/2}\right] = \frac{g\left(m_{a} - m_{r}\right)}{2\pi R_{ef}} + 2\gamma_{L}$$
(8)

3. RESULTS AND DISCUSSION

Results of experiments from advancing and receding modes of n-alkanes and other probe liquids in the porous beds of alumina are listed in Table 2.

As can be seen the reproducibility of imbibition and drainage experiments of probe liquids into the powder beds were very good and in all cases with standard deviation less than 4%. The effective radius of alumina porous bed was calculated from the n-alkane weights in the powder in the receding mode by using Eq. (6). These results are presented in Table 2. Moreover, the effective radius was similar for all tested alumina powders packed in the tips.

Tab. 2. Weights of probe liquids in the porous beds of alumina for advancing (A) and receding (R) modes in mg, and total effective radius for particular samples in cm/0.4g

Al ₂ O ₃ sample	Mode	n-Nonane	n-Decane	n-Dodecane	R _{av.}	Diiodomethane	Water	Formamide
Al ₂ O ₃	A	172.6±0.1	175.9±4.3	181.7±0.2		788.9±7.3	240.9±9.8	282.1±4.8
	R	186.7±1.8	189.0±2.3	196.3±1.2	1.240 ± 0.031	870.6±15.2	268.4±3.7	305.4 ± 3.0
$Al_2O_3 + MF$	A	168.2±2.6	175.0±1.4	180.5±2.1		777.1±9.6	239.8±0.1	264.2±2.4
	R	184.8±2.8	186.4±0.4	198.4±0.1	1.234±0.022	865.8±5.7	260.6±2.1	299.2±4.7
Al ₂ O ₃ /Ca ²⁺	A	169.3±0.1	179.5±2.6	178.5±3.4		780.3±5.5	237.8±7.6	267.7±3.4
	R	179.5±2.6	182.6±5.0	191.3±3.2	1.190±0.038	851.1±6.1	252.1±3.9	286.2±4.4
$Al_2O_3/Ca^{2+} +$	A	168.4±3.3	174.7±2.2	176.9±2.1		778.8±9.1	234.5±0.9	254.2±9.9
MF	R	179.7±1.5	186.6±4.5	191.9±4.5	1.210±0.025	869.5±9.3	245.6±3.9	288.6±4.4
Al ₂ O ₃ /La ³⁺	A	174.6±1.9	177.4±2.0	180.9±1.8		781.1±12.7	239.7±1.7	261.4±1.7
	R	183.2±2.0	194.5±1.9	194.4±0.3	1.240 ± 0.040	868.6±1.4	250.1±1.9	292.1±2.4
$Al_2O_3/La^{3+} +$	A	168.3±2.6	171.0±2.7	175.7±4.2		786.3±3.1	240.8±3.4	263.5±9.2
MF	R	179.1±0.2	188.5±2.5	187.0±6.5	1.200±0.050	853.0±13.0	251.7±5.9	296.4±1.8

These radii were used for calculation of the surface free energy components from imbibition and filtration experiments with diiodomethane, water and formamide for bare alumina and modified with Ca²⁺ and La³⁺ ions in the absence and presence of magnetic field. The results are collected in Table 3 and Figure 1.

Tab. 3. Surface free energy components of alumina (mJ/m²)

Surface	$\gamma_{\mathrm{S}}^{\mathrm{LW}}$		$\gamma_{\scriptscriptstyle m S}^-$		$\gamma_{\mathrm{S}}^{\scriptscriptstyle +}$		$\gamma_{ m S}^{ m AB}$	
	no MF	+ MF	no MF	+ MF	no MF	+ MF	no MF	+ MF
Al_2O_3	41.0	40.2	52.9	56.6	0.78	0.59	12.9	11.6
Al_2O_3/Ca^{2+}	41.9	39.8	55.2	59.0	0.66	0.58	12.1	11.7
Al_2O_3/La^{3+}	40.4	42.5	58.4	59.1	0.59	0.46	11.7	10.4

In calculations of the surface free energy components for the systems, in which the magnetic field was present it was assumed that the field does not change the components of surface tension of the probe liquids. This assumption results from the fact that there is the lack of possibilities to measure those values when the magnetic field influences the studied systems. In all the cases the same probe liquids were used, thus such approximation should not influence the calculations.

As can be seen in Table 3 the apolar Lifshitz-van der Waals component of bare alumina equals 41 mJ/m² and is practically the same as γ_s^{LW} of alumina determined by others using the same method [10,14] and thin layer wicking method [16–18] (see Tab. 4, where the literature values of surface free energy components of other samples alumina are listed).

Tab. 4. The literature values of surface free energy components of alumina (mJ/m²)

Method	$\gamma_{\rm S}^{\rm LW}$	$\gamma_{ m S}^-$	$\gamma_{\rm S}^+$	$\gamma_{\rm S}^{\rm AB}$
IDM [10]	41.7±0.3	56.8	0.6	11.7
IDM [14]	40.0	55.0	0.7	12.4
TLWM [16]	40.0	54.4	1.23	16.4
TLWM [17]	41.0	72.4	0.3	9.3
TLWM [18]	43.7	80.5	0.004	1.1
TLWM [19]	31.6	27.2	0.6	8.1
GCM [20]	54.3±3.2	33.3±3.6	40.1±6.0	73.1

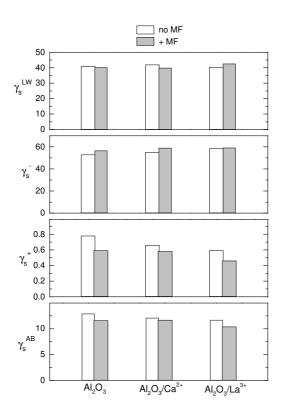


Fig. 1. Surface free energy components of alumina modified with Ca²⁺ and La³⁺ ions in the absence and presence of magnetic field

Both adsorbed Ca^{2+} or La^{3+} ions on alumina surface and treatment the sample with magnetic field did not significantly affect the apolar interactions. From the results (Tab. 3) it appears that alumina shows a distinctly basic character of the surface (γ_S =52.6 mJ/m²), whereas the acid parameter has a small value (γ_S^+ =0.79 mJ/m²). This has also been found for the majority of polar solids and liquids [4,21]. It is seen that the electron-donor and electron-acceptor parameters of bare alumina determined in this paper are very similar to those obtained by others [10,14,16], but differ from the values presented in the papers [17–20]. The presence of adsorbed ions increases the electron-donor parameter and decreases the electron-acceptor one; in effect acid-base interactions practically do not change. The treatment with the magnetic field causes additional small increase in γ_S and decrease in γ_S^+ of Al_2O_3 , Al_2O_3/La^{3+} and Al_2O_3/Ca^{2+} systems in comparison to the corresponding without magnetic field treatment.

The differences in the components might be due to not only the various methods used for their determination but also the samples origin.

Giese et al. [18] obtained lower the values of the surface free energy components for alumina from thin layer wicking method, because they had determined them from contact angles calculated from wicking experiments and Washburn's equation. It is known that determined in this way contact angles are not equal to Young's contact angles of a drop of the same liquid placed on flat surface of the solid [22]. Consequently, the solid surface free energy components calculated using contact angles from Washburn's equation are not the true values.

The results of surface free energy components determined by IDM differ from those obtained by Biliński et al. [20]. Applying the gas chromatography method they found higher electron-acceptor than electron-donor interactions of basic alumina surface (Tab. 4). Similar results, i.e. the prevailing electronacceptor interactions, were also found for silica [23]. The differences in the values of solid surface free energy components may be also explained in terms of some real differences in the surface properties, resulting from specificity of the measurement conditions related to particular method. For example prior to gas adsorption measurements the column is always conditioned with dry carrier gas and physically adsorbed water is removed. This means, that the solid surface properties investigated by gas chromatography method are not necessarily the same as those investigated by IDM, TLWM or contact angle measurements. The problem seems relies on the presence of a water layer adsorbed under the measurement condition, hence, the obtained values of surface free energy components by IDM should be considered in terms of water molecules presence on the surface. According to Grivtsov et al. [24] adsorption of water on the surface silanol groups may take the place in two different ways, the hydrogen bond may by formed between an O atom from the surface silanol and H atom from a water molecule, or between H atom from silanol and an O atom of water molecule.

Considering the first, more probable model of hydrogen bonding formation one may conclude that the alumina surface becomes more basic (electron-donor). However, the second model cannot be entirely excluded. Assuming that the electron-donor interaction fully results from the presence of hydroxyl groups on alumina surface and their ability to form hydrogen bonding between oxygen atom from OH group and hydrogen atom from water [24], then taking the energy of hydrogen bonding and γ_S the number of OH groups per 1 nm² of the surface area can be estimated. The energy of hydrogen bonding interaction is higher than that due to van der Waals forces. The commonly accepted values of the energy of hydrogen bonding vary between 10 and 40 kJ mol⁻¹ [24].

The value of 10 kJ mol $^{-1}$ corresponds to $1.66\cdot 10^{-20}$ J per single bonding. For bare alumina (γ_s =52.9 mJ/m 2 , i.e. $5.84\cdot 10^{-20}$ J/nm 2) the surface concentration of hydroxyl group would amount to 3.2 OH groups per 1 nm 2 , which interact as electron-donor. On the other hand, considering the electron-acceptor interaction (γ_s =0.79 mJ/m 2) the calculation gives 0.05 hydroxyl group/nm 2 or 5/100nm 2 . The presence of Ca $^{2+}$ and La $^{3+}$ ions on the alumina surface increases the electron-donor parameter and thereby the number of OH groups (Fig. 2). This can be interpreted in fact that the adsorbed ions hydrolysed.

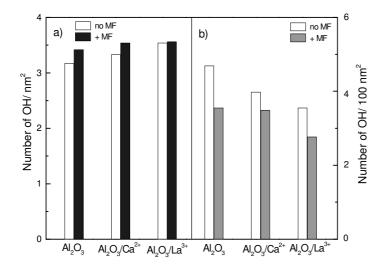


Fig. 2. The surface concentration of hydroxyl groups on alumina surface which interact as electron-donor (a) and/or electron-acceptor (b)

Although Al_2O_3 surface was modified by different ions the effect of the magnetic field on electron-donor interaction is similar. In our earlier studies we concluded that magnetic field effects were, among other things, connected with changes in water molecules structure in the hydration shell of ions present in the system [26,27]. Similar effect of magnetic field on γ_S observed in the presence of Ca^{2+} and La^{3+} may result from the fact, that the hydrated radii of these ions are similar (0.171 and 0.203 nm, respectively) and the mean time of water molecule residing in the cation hydration shell is of the same order $(3\cdot10^{-9})$ and $5\cdot10^{-9}$ s), despite of different hydration number, i.e. water molecules present in the first hydration shell of the cations (7.2 and 10.3) [28].

4. CONCLUSIONS

From obtained results it may be concluded that the imbibition drainage method is useful for determination of surface free energy components of powdered solids for which it is difficult to measure contact angles. The surface free energy components of tested alumina agree well with the values determined by thin layer wicking method for similar materials but differ from those obtained by the gas chromatography method. The discrepancies in the components might be not only different experimental methods used for their determination, but also the sample origin. The presence of deposited ions and magnetic field influence physicochemical properties of alumina surface and cause changes in the surface free energy components. The obtained values should be considered taking into account water molecules adsorbed on the alumina surface and hence the magnetic field effect may be due to some changes in the water hydrating structure.

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He published over 120 papers in professional journals and books. He was also a member of the International Advisory Committee of 9^{th} and 10^{th} International Symposium on Surfactants in Solution (1992, Varna, Bulgaria, and 1994, Caracas, Venezuela) and 1^{st} International Congress on Adhesion Science and Technology (1995, Amsterdam, The Netherlands). At present he is a member of the Committee of 13^{th} International Symposium on Surfactants in Solution, to be held in Gainesville, Florida, June 11–16, 2000.

Main scientific interest: Interfacial phenomena at solid-liquid, solid-gas, and liquid-liquid interfaces, electrochemical phenomena, surface and interfacial free energy, its components and their experimental determination, suspensions, emulsions, stability of the dispersed systems, effects of the external fields (high frequency electric and magnetic) on the properties of the dispersed systems, adhesion, wetting, flotation, stability of the dispersed systems.