Investigation of the surface properties of quartz-based dispersed materials

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Abstract. Researched quartz dispersive materials (Ukraine): mineral - quartzite of the Ovruch deposit and quartz sand of the Novovodolazsky deposit and products of processing of quartz minerals - dinas and marshelite. The mineral composition, surface morphology, dispersion and specific surface area were experimentally studied. The structure of the hydroxyl-hydrate surface layer was studied by thermogravimetric (TG) analysis and differential thermal analysis (DTA). The number and acid-base characteristics of surfaces active centers were investigated by the methods of pK\(_a\)-metric and pH-metric. It is established that the mechanisms of physical and chemical adsorption of water molecules on the surface of all quartz dispersed materials are identical and do not depend on the mineral nature of impurities. It is shown that the thickness of the surface hydroxyl-hydrate layer of quartz dispersed materials is determined by the amount of chemically adsorbed water, i.e. the thickness of the hydroxyl layer and depends on the nature of the associated minerals. It is established that the acid-base surface characteristic of quartz materials significantly depends on the chemical nature of the accompanying minerals and does not depend on the thickness of the surface hydroxyl-hydrate layer.

1. Introduction and literature review

Quartz dispersed materials are widely used in various fields: to create composite materials for various purposes, as adsorbents for purification of water and other liquids, as abrasives, etc. [1-4]. Due to many properties, composite materials based on polymers and quartz fillers are effectively used in the construction and restoration of roads, bridges, hydraulic structures, residential buildings [5-9]. In some cases, the operational and physical and mechanical properties of such materials are not inferior to known materials - concrete, stone, metals, wood. The presence of quartz disperse fillers provides strength to mechanical loads [4, 7], resistance to water and other aggressive environments [8, 9], resistance to high temperatures [4, 9-11], thermal insulation, sound and vibration protection properties [5, 6, 8, 12]. The use of inorganic materials, including quartz, is due to the chemical nature and acid-base properties of the surface of the dispersed particles. This determines the efficiency and mechanisms of interaction of the surface of dispersed materials with the polymer.

2. Literature review

The high-energy surface of dispersed inorganic materials is characterized by the presence of a layer of surfaces active centers (SAC) [13-18], which, in terms of acid-base theory, can be divided into Lewis centers and Bransted centers. It is established that the surfaces of solid oxides are characterized by a wide range of acid-base SACs of alkaline (MeO\(^-\), O\(^{2-}\), OH\(^{-}\)), acid (Me\(^{n+}\), OH\(^{n+}\)), as well as neutral [15, 16]. The acidity function of SAC can vary widely. For example, the authors of the study [17] found that on the surface of quartz SiO\(_2\) there are acid (pK\(_a\) = 2.5), neutral (pK\(_a\) = 6.4) and basic (pK\(_a\) = 8.8) silanol groups ≡SiOH, negatively charged oxygen atoms –O\(^2\) (pK\(_a\) = −0.2) and positively charged silicon atoms Si\(^{n+}\) (pK\(_a\) = 14.2), as well as siloxane groups Si–O–Si. In [19], the acid-base equilibrium on the SiO\(_2\) surface was investigated using quantum chemical modeling of PAC. Quantum chemical models of anhydrous and hydrated SACs are proposed, the structural formulas of which include the...
Central element of the Si4 + crystal lattice with coordination numbers 4 and 6, oxygen atoms, OH-groups and water molecules. Anhydrous isolated centers contain one, two (geminal center) or three OH-groups and are connected to the central element. The vicinal centers contain two central elements and two OH-groups connected by a hydrogen bond. In addition to the three types of anhydrous centers, the subject of the study were hydrated centers that contain from one to five water molecules and are attached to the anhydrous center in accordance with the known mechanism of hydration of surface OH-groups [20-23]. It is shown [19] that on the surface of SiO2, regardless of the coordination number of Si4+, the smallest parameters of pKa and, accordingly, the largest acidic properties are characterized by vicinal anhydrous centers Si2O5(OH)2 (pKa = 9.2) and Si2O6(OH)2 (pKa = 11.9). Centers with the coordination number of the Central element 6 show less acidity than centers with the coordination number 4. The most basic are characterized by isolated active centers with one OH-group SiO2OH (pKa = 11.9), SiO2OH (pKa = 13.1). As a result of calculations, it was found that the acidity of the isolated centers increases in direct proportion to the increase in the number of OH-groups. It is established that with the increase in the number of adsorbed water molecules, the acidity of all types of SAC increases. The identical character of growth of acidity of SAC at increase in thickness of both hydroxyl, and hydration layers of a surface is revealed. The calculated values of the free Gibbs energy of the processes of deprotonation of the centers are positive and vary from 52.6 to 74.7 kJ / mol.

Determining the acid-base characteristics of the active centers of the surface layer of dispersed materials is an important scientific task. For this purpose, methods are used that allow to determine the number and acid-base parameters of SAC experimentally. For example, methods of IR spectroscopy [24], gas adsorption [20, 26] or color indicators [25, 27-29]. In [15, 16, 23, 30-33] the method of potentiometric titration of suspensions is used. Experimental methods are informative, but time-consuming and do not allow to determine the chemical nature of active centers. To elucidate the chemical nature of SAC and study the protolytic equilibrium on the surface of SiO2 [19, 21] and in other mixed and pure oxides [22], the authors propose a theoretical method of quantum chemical modeling.

The spectrum of SAC of different chemical structure forms the surface hydroxyl-hydrate layer (SHHL) of dispersed inorganic material, which is characterized by a certain thickness and acid-base properties [13-16]. SHHL of inorganic materials consists of two main layers. The lower hydroxyl dense layer is formed from water molecules chemically bound to the surface. The upper is a less dense hydrate layer, which consists of adsorbed water molecules on the surface of the lower hydroxyl layer due to hydrogen bonds. The presence of SHHL provides physicochemical interactions that occur on the surface of dispersed materials. In [15, 16] the structure and acid-base properties of SHHL of clay and oxide inorganic materials were studied. It was found that the acid-base surface properties of oxide materials based on TiO2, Al2O3, CaO, Fe2O3 correlate with the acid-base properties and nature of oxides and do not depend on the thickness of SHHL. It has been shown that the number of chemically and physically bound water molecules does not affect the acid-base properties of the surface of oxide materials [16]. It was also found that the determining factor in the acid-base surface properties of clay fillers is the mineral composition. The thickness of the SHHL affects the acidity of the surface to a small extent. It was found that clay fillers, which include montmorillonite as the dominant mineral, are characterized by higher acidity than fillers consisting of kaolinite. In mixed clay fillers, the acid-base properties of the surface depend on the chemical nature of impurities and associated minerals [15]. Data on the surface of quartz dispersed materials in modern studies are rare, they are unsystematized and incomplete.

Thus, the purpose of this scientific work is to study the properties of SAC and SHHL of the most popular quartz dispersed materials. This will create opportunities to predict the nature and mechanism of interaction of the surface of quartz dispersed materials on the phase separation surface and will create the preconditions for their effective use in practice.
3. Materials of research
As materials for research the dispersed materials on the basis of quartz (Ukraine) are chosen: mineral – quartzite of the Ovruch deposit (QO) and quartz sand of the Novovodolazsky deposit (QN) and products of processing of quartz minerals – dinas (DS) and marshalite (MT). Quartzite of the Ovruch deposit is a gray-yellow mineral powder, the main raw material for making dinas and ferrosilicon in Ukraine and Eastern Europe. Dense, hard, fine-grained rocks, which consist mainly of β-quartz grains. Quartz sand of the Novovodolazhsky field of the PK-015-3 brand is a powdery white material. Dinas is a light gray powdery material, a product of quartzite firing with the addition of limestone and iron scale to increase the content of CaO and Fe₂O₃ in the resulting product. The firing process is carried out at temperatures of 1400–1750 °C. Marshalite - produced brands "A" and "B", a natural dusty quartz material of gray-white color, the main mineral of which is β-quartz SiO₂ with small admixtures of chalcedony, opal, carbonates and clay minerals.

4. Methods of research
The mineral composition of quartz dispersed materials was studied on an X-ray diffractometer DRON-2 (Russia). Particle size and surface morphology were determined using a scanning electron microscope brand JSM-6390LV (Japan). The specific surface was determined by the BET method (Brunauer, Emmett, Teller). Thermogravimetric analysis and differential thermal analysis using SDT Q600 and Instruments (USA) thermal analyzer were used to study SHHL.

Physic-chemical experimental methods – pK-metric and pH-metric – were used to study the acid-base properties of SAC and SHHL of quartz dispersed materials. Both methods are described in detail in studies [15, 16].

5. Study of mineral composition, size particles and surface area of quartz dispersed materials
The results of the study of mineral composition, size particles and surface area of quartz dispersed materials are presented in Table 1 and on Figures 1-4.

| Quartz dispersed material | Mineral composition                        | Surface area (m²/g) | Preferred particle size (μm) |
|--------------------------|-------------------------------------------|---------------------|-----------------------------|
| QO                       | β-quartz SiO₂•                             | 3.03                | 10–20                       |
|                           | Montmorillonite                           |                     |                             |
|                           | (Al,Mg)₂(OH)₂[Si₂O₁₀]nH₂O⁺               |                     |                             |
|                           | Kaolinite Al₂Si₂O₅(OH)₄⁺                   |                     |                             |
|                           | Albite Na₂O·Al₂O₃·6SiO₂⁺                   |                     |                             |
|                           | Orthoclase K₂O·Al₂O₃·6SiO₂⁺                |                     |                             |
|                           | β-quartz SiO₂•                             |                     |                             |
| QN                       | Montmorillonite                           | 2.91                | 10–15                       |
|                           | (Al,Mg)₂(OH)₂[Si₂O₁₀]nH₂O⁺               |                     |                             |
|                           | β-quartz SiO₂•                             |                     |                             |
| DS                       | Cristobalite α-SiO₂•                      | 2.16                | 20–30                       |
|                           | Tridymite SiO₂•                           |                     |                             |
|                           | β-quartz SiO₂•                             |                     |                             |
| MT                       | Microline K₂O·Al₂O₃·6SiO₂⁺                 | 2.75                | 5–10                        |

Remarks: ● – basic mineral phase; + - side mineral phase; *- traces.

X-ray phase analysis of QO shows the presence in addition to the main phase of β-quartz impurities of accessory minerals: potassium and sodium feldspar (orthoclase and albite). Kaolinite and montmorillonite are identified in trace amounts. Isometric particles of irregularly shaped QO have a cancerous fracture, the surface without porous. Dispersion exceeding 10–20 μm, large fraction (40–
50 μm) is present in the sample up to 10%. The fine fraction is present in small quantities, which mostly consists of particles smaller than 5 μm (Figure 1).

![Microphotographs for quartzite of the Ovruch deposit (QO).](image)

Figure 1. Microphotographs for quartzite of the Ovruch deposit (QO).

The QN diffraction pattern is represented by a set of pronounced diffraction maxima corresponding to β-quartz SiO₂ of high crystallinity. Weak peaks of montmorillonite indicate a low content of clay component in the sand sample, which is confirmed by scanning microscopy: microphotographs clearly visible fragments of quartz grains with a smooth without porous surface. Clay minerals are not observed, respectively, we can talk about sufficient purity of sand, consisting of SiO₂ crystallized in the tetragonal system (β-quartz). The preferred dispersion of the powder is 10-15 μm. A sufficient fraction (2–4 μm) is present in sufficient quantities, and large particles with a size of 25–30 μm are found (Figure 2).

According to the results of X-ray phase analysis, in addition to low-temperature tridymite, unregenerated quartz and weakly structured cristobalite are present in the DS sample. The photomicrographs show thin needle-like crystals of tridymite grown on isometric quartz grains and bound to a weakly structured cristobalite. The preferred dispersion of DS is 20–30 μm. The size of the crystals of the tridymite phase varies in the range of 0.15–0.3 μm (Figure 3).

Highly dispersed MT powder with a predominant dispersion of 5–10 μm was used for the study. The size of large particles is 50-60 microns. The thin fraction of 0.2–2 μm is in small quantities (Figure 4). According to the results of X-ray phase analysis, the main crystalline phase of the powder is β-quartz with a high degree of crystallinity. A microcline is present in the sample as trace impurities.
Figure 2. Microphotographs for quartz sand of the Novovodolazsky deposit (QN).

Figure 3. Microphotographs for dinas (DS).
Figure 4. Microphotographs for marshellite (MT).

As can be seen from the results presented in Table 1, the specific surface area and particle size of mineral quartz dispersed materials QO (3.03 m²/g and 10–20 μm) and QN (2.91 m²/g and 10–15 μm) are identical. Heat treatment of quartzite (firing) produces a DS material with a smaller specific surface area (2.16 m²/g) and, due to sintering, with a larger particle size (20–30 μm). Machining of quartzite (grinding) produces an MT material with a smaller particle size (5–10 μm) and a slightly smaller specific surface area (2.75 m²/g).

6. Study of the dynamics of mass loss of quartz dispersed materials

In Table 2 shows the dynamics of mass loss of quartz materials in the process of increasing temperature.

| Quartz dispersed material | Weight loss of the materials in the temperature range (%) |
|--------------------------|----------------------------------------------------------|
|                          | 0–150°C | 150–300°C | 300–600°C | 600–900°C |
| QO                       | 0.3     | 0.1       | 0.9       | 0.5       |
| QN                       | 0.3     | 0.2       | 0.5       | 0.2       |
| DS                       | 1.0     | 0.6       | 1.6       | 0.2       |
| MT                       | 0.3     | 0.2       | 0.6       | 0.3       |

From the results presented in Table 2 it follows that the processes of weight loss when heated of all quartz materials occur identically. Thus, the most intense weight loss in all cases is observed in the temperature range of 300–600 °C. In the temperature range up to 150 °C in materials QO, QN and MT weight loss has the same value (0.3%). This indicates that the surface of these materials contains approximately the same amount of physically adsorbed water. The DS surface is characterized by three times more physically adsorbed water (weight loss 1%). Obviously, this is due to the fact that DS is a product of quartzite firing. Thus, it can be argued that the mechanisms of physical and chemical adsorption of water molecules on the surface of all quartz dispersed materials are identical and do not
depend on the mineral nature of impurities. Obviously, the adsorption mechanism is affected only by the method (thermal or mechanical) processing of the material.

It should be noted that at temperatures of 0–300 °C there are processes of dehydration of the surface of the dispersed material, i.e. the loss of physically adsorbed water molecules (hydrate layer). At temperatures of 300–900 °C, the processes of surface dehydroxylation occur, i.e. the loss of chemically bound water molecules (hydroxyl layer). The total weight loss in the entire temperature range, i.e. up to 900 °C, determines the thickness of SHHL. Thus, the thickness of the hydrated surface layer of quartz dispersed materials increases in a row:

$$\text{QO} (0.4\%) < \text{QN} (0.5\%) < \text{MT} (0.5\%) < \text{DS} (1.6\%).$$

(1)

The thickness of the hydroxyl layer of quartz dispersed materials increases in a row:

$$\text{QN} (0.7\%) < \text{MT} (0.9\%) < \text{QO} (1.4\%) < \text{DS} (1.8\%).$$

(2)

The thickness of SHHL of quartz dispersed materials increases in a row:

$$\text{QN} (1.2\%) < \text{MT} (1.4\%) < \text{QO} (1.8\%) < \text{DS} (3.4\%).$$

(3)

As can be seen from the obtained series (1), the thickness of the surface hydrate layer depends only on the method of processing quartz dispersed materials and, obviously, does not depend on the mineral composition. Series (2) indicates that the thickness of the surface hydroxyl layer is 2–3 times greater than the thickness of the hydrate layer for all quartz materials. The amount of chemically adsorbed water depends on the mineral nature of the impurities. The presence of clay minerals montmorillonite and kaolinite determines the greatest thickness of the hydroxyl layer QO. As can be seen from row (3), the thickness of SHHL quartz dispersed materials is determined by the amount of chemically adsorbed water, i.e. the thickness of the hydroxyl layer and, accordingly, also depends on the nature of the associated minerals.

7. Study of pK-metric and pH-metric methods for quartz dispersed materials

According to the results of the pK-metric method, the following indicators were calculated: the number of acid SACs ($\sum q_A$); the number of alkaline SAC ($\sum q_B$); the total number of SAC ($\sum q = \sum q_A + \sum q_B$); integral index ($Q = \frac{\sum q_A}{\sum q_B}$), which characterizes the surface of quartz material as neutral ($Q \approx 1$), acidic ($Q > 1$) or alkaline ($Q < 1$). According to the results of the pH-metric method, the following indicators were calculated: equilibrium value of the hydrogen index of the aqueous suspension of quartz material ($pH_{eq susp}$); the value of the isoadsorption point of the aqueous suspension of quartz material, at which there is no mass transfer of ions from the solution to the surface of the solid ($pH_{IP} = pH_0 + (pH_{eq \text{ susp}} - pH_0) \cdot (pH_0 - 6)$); an indicator indicating the width of the pK SAC range on the surface of the quartz material ($\Delta pH = pH_{IP} - pH_{eq \text{ susp}}$); an indicator that characterizes the duration of the equilibrium state in the aqueous suspension of quartz material ($t^{eq}$). The results of pK-metric and pH-metric experimental studies of quartz dispersed materials are presented in Table 3 and Table 4.

The presented results show that the data obtained by different methods (pK-metric and pH-metric) correlate with each other. Thus, according to the results of pK-metric analysis (table 3), the alkalinity of quartz materials increases in a number:

$$\text{QN}(Q \approx 0.80) < \text{QO}(Q \approx 0.61) < \text{DS}(Q \approx 0.35) < \text{MT}(Q \approx 0.28).$$

(4)

According to the results of pH-metric analysis (table 4), the alkalinity of the surface of quartz materials, in accordance with the value of $pH_{IP}$, increases in a number:

$$\text{QN}(7.16) < \text{QO}(8.94) < \text{DS}(9.51) < \text{MT}(9.74).$$

(5)

From the results presented in table. 1, 3 and 4 it follows that the acid-base surface characteristics of quartz materials significantly depend on the chemical nature of the accompanying minerals. The
highest acidity is characterized by the surface QN (Q=0.80; pH_{IP}=7.16), which contains montmorillonite. This is confirmed by the known fact [15] that the presence of montmorillonite determines the acidic properties of the clay dispersed material. On the QN surface, in addition to Bronsted's neutral SACs, strongly acidic SACs with pK≈0.5–1.5 (possibly partly Lewis character) and alkaline SACs with pK≈9–10 were detected. The weakly alkaline nature of the QO surface (Q=0.61; pH_{IP}=8.94) is due to the fact that in the presence of montmorillonite the composition includes impurities of kaolinite. Apparently, this is due to the fact that the surface of dispersed kaolinite is known to have an alkaline reaction [15]. The surfaces of DS (Q≈0.35; pH_{IP}=9.51) and MT (Q≈0.28; pH_{IP}=9.74) differ in the greatest alkalinity. This is due to the presence on the surface of a large amount of SAC with pK≈9–11.

### Table 3. Results of study of quartz materials pK-metric method.

| Quartz dispersed materials | $\sum q_A \cdot 10^{12}$ (1/sm$^2$) | $\sum q_B \cdot 10^{12}$ (1/sm$^2$) | $\sum q \cdot 10^{12}$ (1/sm$^2$) | $Q = \frac{\sum q_A}{\sum q_B}$ |
|---------------------------|-------------------------------------|-------------------------------------|-------------------------------------|--------------------------------|
| QO                        | 48.51                               | 80.14                               | 128.65                              | 0.61                           |
| QN                        | 152.23                              | 189.52                              | 341.75                              | 0.80                           |
| DS                        | 79.13                               | 226.09                              | 305.22                              | 0.35                           |
| MT                        | 45.74                               | 163.36                              | 209.10                              | 0.28                           |

### Table 4. Results of study of quartz materials pH-metric method.

| Quartz dispersed materials | pH$_0$ for distilled water | pH$_{eq,susp}$ | pH$_{IP}$ | $\Delta$ pH | $t_{eq}$ (s) |
|---------------------------|---------------------------|----------------|----------|-------------|--------------|
| QO                        | 7.45                      | 8.48           | 8.94     | 0.46        | 360          |
| QN                        | 7.25                      | 7.18           | 7.16     | 0.02        | 40           |
| DS                        | 7.50                      | 8.84           | 9.51     | 0.67        | 120          |
| MT                        | 7.10                      | 9.50           | 9.74     | 0.24        | 300          |

A comparison of all the obtained regularities (1–5) allows us to state that the acid-base surface properties of quartz materials do not depend on the SHHL thickness.

8. Conclusions

The results of the study allow us to formulate the following conclusions. For the most popular quartz dispersed materials quartzite of the Ovruch deposit (QO), quartz sand of the Novovodolazsky deposit (QN), dinas (DS) and marshalite (MT), the mineral composition, surface morphology, predominant size and specific surface area of the particles are determined. The structure of the SHHL of quartz materials was investigated using thermogravimetric (TG) and differential thermal analysis (DTA). The number and acid-base characteristics of SACs were studied by pK- and pH-metric.

It is established that the mechanisms of physical and chemical adsorption of water molecules on the surface of all quartz dispersed materials are identical and do not depend on the mineral nature of impurities. Obviously, the mechanism of adsorption of water molecules is affected only by the method (thermal or mechanical) treatment of the dispersed material. It is also found that the thickness of the surface hydrate layer depends only on the method of processing quartz dispersed materials and does not depend on the mineral composition. It is shown that the thickness of the surface hydroxyl layer is 2-3 times greater than the thickness of the hydrate layer for all quartz materials, and the amount of chemically adsorbed water depends on the mineral nature of the impurities. The presence of clay minerals montmorillonite and kaolinite determines the greatest thickness of the hydroxyl layer of quartzite. The thickness of SHHL quartz dispersed materials is determined by the amount of chemically adsorbed water, i.e. the thickness of the hydroxyl layer and, accordingly, also depends on the nature of the associated minerals.
It is established that the acid-base characteristic of the surface of quartz materials significantly depends on the chemical nature of the accompanying minerals. It has been experimentally shown that the surface QN, which contains montmorillonite, is characterized by the highest acidity. On the QN surface, in addition to neutral SACs, strong acid SACs with pH≈0.5–1.5 and alkaline SACs with pH≈9–10 were detected. The weakly alkaline nature of the QO surface is due to the fact that in the presence of montmorillonite the composition includes impurities of kaolinite. The DS and MT surfaces differ in the highest alkalinity due to the presence of a large amount of SAC with pH≈9–11 on the surface. It was found that the acid-base surface properties of quartz dispersed materials do not depend on the thickness of SHHL.

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