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The significance of the difference in the point of zero charge between rutile and anatase

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Abstract

The points of zero charge (PZC) of titanium dioxide reported in the literature range from 2 to 8.9. A set of 138 PZC of titanium dioxide was used to explore the effect of the crystalline structure on the PZC. The average and median PZC at pH 5.6 and 5.8, respectively, was found when the entire data set was taken into account. The PZC of anatase (31 entries, average and median 5.9 and 6, respectively) is slightly higher than that of rutile (49 entries, average and median 5.4 and 5.5, respectively), and the difference between the polymorphs corresponds to half of a standard deviation in each set of PZC.

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Keywords: Point of zero charge; Adsorption; Isoelectric point; Rutile; Anatase

Contents

1. Introduction

Titanium dioxide is an ideal model adsorbent for studies of the relationship

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Table 1
The physical properties of rutile and anatase

Property	Rutile	Anatase
Crystallographic system	Tetragonal	Tetragonal
(1) System number	136	141
(2) Schoenflies	D4h^{14}	D4h^{19}
(3) Hermann–Mauguin	P4$_2$/mnm	I4$_1$/amd
Specific density (kg m^{-3})	4130	4060
G^0 (kJ mol^{-1})	−960	−956
H^0 (kJ mol^{-1})	−945	−941
C_p (J mol^{-1} K^{-1})	55.1	54.02

between the surface charging and adsorption. Its solubility is negligible, and its point of zero charge (PZC) in the middle of the pH scale [1] makes it possible to study adsorption on positively and negatively charged surface of titanium dioxide over a broad range of pH and ionic strength. Titanium dioxide has numerous applications, e.g. as a pigment and as a catalyst support, and its surface properties have been extensively studied. The PZC of titanium dioxide (and other metal oxides) published before 1965 were compiled by Parks [2]. The PZC of titanium dioxide published before 1976 were compiled by Parfitt [3]. The PZC of titanium dioxide (and other metal oxides) published between 1966 and 1999 were compiled by Kosmulski [1]. A few other compilations of the PZC of titanium dioxide have been published (cf. Ref. [4] for references and discussion).

Rutile (one of crystallographic forms of titania) is thermodynamically stable at room temperature, and anatase (another crystallographic form) is kinetically stable, i.e. its transformation into rutile at room temperature is so slow, that the transformation practically does not occur. Transformation of macroscopic specimens of anatase into rutile reaches a measurable speed at $T>800$ °C [5,6]. With nanosized anatase, the transformation reaches a measurable speed at $T>400$ °C [7,8]. The rate of transformation is severely affected by doping of anatase with various metals [9,10], and even by pretreatment of anatase with butanol [11], yet in all studies the rate of transformation at $T<400$ °C was negligible.

The studies of surface charging of titania have been carried out with rutile, with anatase, and with mixtures thereof. There is also another form of titania, namely, brookite, but to the best knowledge of the present author surface charging of brookite has not been studied. The physical properties of rutile and anatase are compared in Table 1.

The dominant faces in the morphology of rutile are (1 1 0), (1 0 0) and (1 0 1), and for anatase (0 1 1) and (0 0 1), respectively, as found experimentally (with macroscopic specimens) and confirmed by atomistic simulation [12]. The analysis of the effect of the surface structure of rutile on the surface site density and surface acidity was carried out by Koretsky et al. [13]. Hiemstra et al. [14] analyzed the effect of the surface structure on the PZC of rutile and anatase in terms of their modified MUSIC model. Their theoretical calculations resulted in PZC at pH 6 for the both polymorphs.

The empirical PZC of titania published in the literature show certain degree of scatter. The difference in the surface properties between anatase and rutile was considered as a possible reason for these discrepancies [15], and anatase has apparently a higher PZC. This hypothesis will be examined in the present paper. The effect of the choice of the experimental method on the PZC of titania will be also explored.

2. Literature survey

The present survey of PZC (and isoelectric points (IEP)) of titanium dioxide is confined to the results published after 1965, and obtained in the presence of inert electrolytes, i.e. alkali nitrates V, chlorates VII and halides at concentrations up to 0.1 mol dm^{-3}. The studies of specific adsorption on titanium dioxide and its effects on the PZC and IEP is beyond the scope of the present paper and it has been discussed in detail elsewhere [1]. The literature was updated untill April 2002 [16–148].

Identical PZC republished in two or more papers by the same authors, are treated as single entry in the present analysis. On the other hand, the PZC of different samples are treated as separate entries also when they were reported in one paper. The temperature effect on the PZC of titanium oxide is rather insignificant (~0.01 pH unit per 1 K), then, the contribution of the temperature effect to the scatter of the published results is negligible (the results analyzed in the present study relate to different temperatures, usually from 20 to 30 °C, and in some publications the temperature was not reported or even not controlled). The PZC at the temperature closest to 25 °C was chosen from publications reporting the PZC at various temperatures. A few publications [149–151] report only negative electrokinetic potentials (this would suggest the IEP of titania at very low pH), but such results were not used in the present analysis.

3. Materials

The samples were sorted in terms of their crystalline structure into the following three categories:

- anatase
- rutile
- other than anatase or rutile or mixture of different forms or structure unknown/not reported.

4. Experimental methods

The PZC of TiO_2 can be determined by means of potentiometric titration of by means of electrokinetic methods. The principles of these methods and their different

Table 2
The PZC of titanium dioxide as the function of the crystalline structure

	Entries	Median	Average	Standard deviation
Anatase	31	6	5.88	0.95
Rutile	49	5.5	5.36	0.83
Other	58	5.9	5.72	1.28

modifications have been discussed in detail elsewhere [1,152]. A few other methods have been used to determine the PZC, but their significance is limited.

In the presence of inert electrolytes the CIP (crossover point of titration curves of oxide dispersion at different ionic strengths) matches the IEP. Such a pH value is very likely the pristine PZC of TiO_2. The results obtained by means of single method are less reliable. The term 'pristine' PZC is used to distinguish between the PZC obtained in the presence of inert electrolyte and the PZC affected by specific adsorption of anions or cations. Publications with matching CIP and IEP are treated as one category of the PZC in the present analysis.

Many publications report only CIP (no electrokinetic data). The existence of CIP does not prove purity of the sample, namely, CIP occurs also in the presence of specific adsorption [152]. These zero points are considered as the second category of the PZC in this paper. Some other publications report CIP and IEP which do not match. With such discrepancies the CIP is favored over the IEP, i.e. the IEP is ignored in the present analysis and only the CIP is taken into account (the second category of the PZC). Finally, a few papers report the PZC obtained by salt titration method [1] which is equivalent to the CIP, and these results also belong to the second category of the PZC.

Many studies report only the IEP (no titration data). The IEP are divided into two categories, namely, results obtained by means of:

- classical electrokinetic methods (electrophoresis, electroosmosis)
- electroacoustic method.

Mass titration and potentiometric titration at one ionic strength are combined into one category. In these methods the PZC is identified with the natural pH of the dispersion.

The PZC of titania obtained by means of other methods or reported without explicitly stating the experimental method were deliberately neglected in the present analysis.

All PZC were rounded to one-tenth of one pH unit.

5. Results and discussion

The PZC of TiO_2 are presented in Table 2 (sorted by structure) and in Table 3 (sorted by experimental method), and the histogram of the PZC with exception of a few extreme values (three highest and three lowest PZC) is presented in Fig. 1. The total height of the bars in Fig. 1 (black + white) corresponds to all entries. Both

Table 3
The PZC of titanium dioxide as the function of the experimental method

	CIP = IEP	CIP	IEP (classical)	IEP (electroacoustic)	Titration (one ionic strength)	Total
Entries	15	30	70	2	21	138
Median	5.9	6	5.6	4.45	5.9	5.8
Average	5.91	5.86	5.51	4.45	5.6	5.63
Standard deviation	0.42	0.72	1.19	2.05	1.31	1.08

black and (black + white) bars produce nearly Gaussian distributions with the centers about pH 6, which also corresponds to the average and median in the entire data set and in the set of PZC obtained as matching CIP and IEP, and > 70% of all published PZC fall in the pH range 5–7.

5.1. Anatase vs. rutile

The present results do not support the allegation [15] that anatase has substantially higher PZC than rutile. The difference in the average and median PZC between the polymorphs is approximately 0.5 pH unit, i.e. half of the standard deviation. Also,

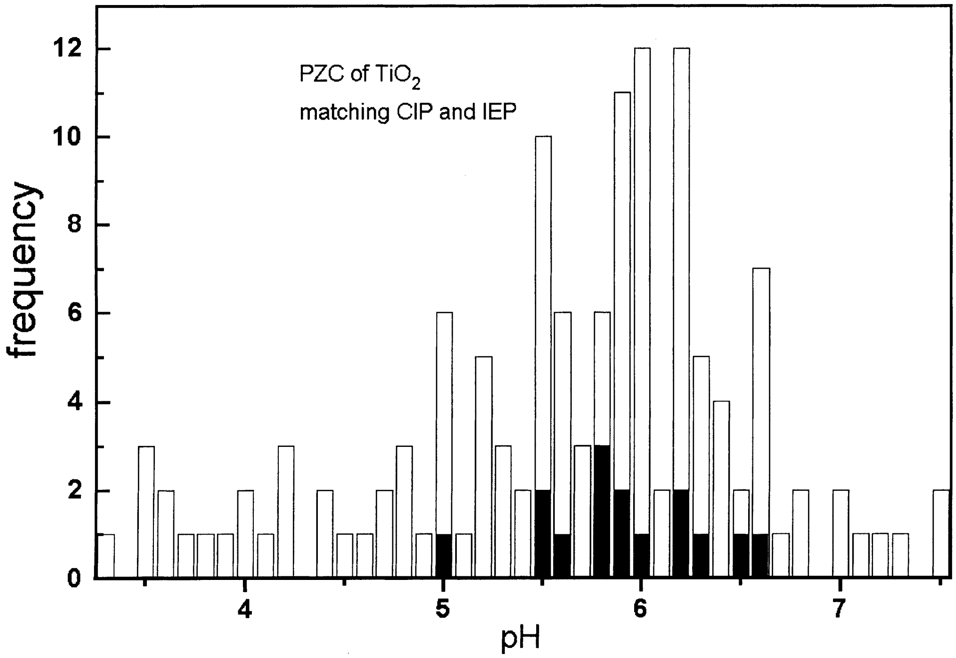

Fig. 1. The distribution of PZC of titanium oxide obtained as matching IEP and CIP (black bars) and all results (white bars).

the analysis of the PZC obtained as matching CIP and IEP (which are more reliable than PZC obtained by means of single method) does not indicate any substantial difference between rutile and anatase. Only two such PZC (pH 5.8 and 6.3) are reported for anatase, while eight PZC reported for rutile are more or less evenly distributed over the pH range 5.5–6.2. The other black bars in Fig. 1 (including the extremes, i.e. pH 5, 6.5 and 6.6) correspond to samples that cannot be classified as pure rutile or anatase.

5.2. Titration versus electrokinetics

All published PZC obtained as matching CIP and IEP (which are more reliable than PZC obtained by means of single method) fall in the pH range 5–6.6. This may suggest that the PZC beyond this range (obtained by means of single method) are due to insufficient purity of samples or other experimental errors.

Most PZC below pH 5 were obtained by electrokinetic methods or by titration at one ionic strength. The low IEP can be explained in terms of the phosphate or other anionic impurities in commercial titanias [19]. The low natural pH of titania dispersions can be explained in terms of traces of acid occluded in pores or in intragrain spaces in titania powders. Interestingly, the discrepancy between the average IEP on the one hand and the average PZC (all results) on the other for titania (the former is lower by 0.4 pH unit) is less significant than the analogous discrepancies for alumina or hematite [1]. Apparently, the ‘silica problem’ (shift in the IEP to low pH due to adsorption of silicate species which are leached out of the glassware at neutral or basic pH) which severely affects the IEP on many metal oxides is rather insignificant for TiO_2. The unusually low average IEP obtained by electroacoustic method (Table 3) is an average of only two values: an unusual IEP at pH 3 [86] and a value of 5.85 [101], which is very close to the average PZC over the entire population. Probably the former low value is due to an experimental error or insufficient purity of the material.

6. Conclusion

The PZC of titania is rather insensitive to the crystallographic structure (anatase versus rutile) or to the choice of experimental method. The ‘recommended’ value is pH 5.9 for the both polymorphs.

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