

K. Siwińska-Stefańska*, A. Krysztafkiewicz*, F. Ciesielczyk*, D. Pauksta*, J. Sójka-Ledakowicz**, T. Jesionowski*

PHYSICOCHEMICAL AND STRUCTURAL PROPERTIES OF TiO₂ PRECIPITATED IN AN EMULSION SYSTEM

Received January 15, 2009; reviewed; accepted May 15, 2009

Precipitation of titania using titanium(IV) sulphate(VI) as the precursor in the emulsion system has been studied. Tytanpol® R-210 of the rutile type structure was used as a standard. The basic physicochemical parameters of the titanium dioxide such as bulk density, the absorption capacity of water, paraffin oil and dibutyl phthalate have been determined along with the moisture content and oil number. Morphology and dispersive characteristics of the titanium dioxide precipitated have been evaluated by the SEM and NIBS techniques. The TiO₂ powders have been also studied by XRD and colorimetric method. In the emulsion system proposed titania of rutile structure has been successfully precipitated. It has been established that the properties of the titanium dioxide obtained depend not only on the composition of the emulsion but also on the amount of the emulsifier applied.

key words: TiO₂, precipitation, emulsion system, rutile and anatase structure, agglomeration

INTRODUCTION

Titania is the most important and most commonly applied inorganic pigment. Chemically it is titanium dioxide (TiO₂) occurring in three polymorphous varieties: tetragonal rutile, anatase and rhombic brookite. Its widespread use is a consequence of

* Poznan University of Technology, Institute of Chemical Technology and Engineering
M. Skłodowskiej-Curie 2 Sq., 60-965 Poznan, Poland
e-mail: Teofil.Jesionowski@put.poznan.pl, phone:+48(61)6653720, fax:+48(61)6653649

** Textile Research Institute (IW), Brzezinska 5/15 St., 92-103 Lodz, Poland

its unique optical and physicochemical properties such as excellent whitening ability, covering power, brightening and lustre ability and resistance to colour change. Excellent pigmentation value is supplemented with the ability to absorb ultraviolet radiation, chemical inertness, high thermal stability and non-toxicity (Wypych 1999, Braun 1992, 1997, Morgans 1990, Buxbaum 1993, The Merck Index 1996, Rentschler 1999, Jesionowski 2002, Doerr 1989, Luo 2007, Chen 2003, Hu 1999).

Titanium dioxide is obtained from natural raw materials such as rock ilmenites, leucogen, ilmenite sands, or from enriched ores such as enriched ilmenites, synthetic rutiles or titanium slags. Titania pigments are produced by two distinct technologies: the sulphate technology and the chloride technology (Wypych 1999, The Merck Index 1996).

The properties of TiO_2 nanoparticles depend on their morphology, size of crystallites and crystallographic structure. A few methods have been proposed for obtaining nanocrystalline particles of titanium dioxide, including hydrolysis (chemical precipitation), microemulsion method (inversed micelles), sol-gel method and hydrothermal crystallisation (Dąbrowski 2001).

TiO_2 nanoparticles have been obtained by the sol-gel method during hydrolysis of titanium precursors (Kim 2002, Liu 2002, Reddy 2001, Manorama 2001, Sugimoto 1987, 1997, 2002a, 2002b, 2003a, 2003b). Usually this process involves hydrolysis of titanium(IV) alkoxides in the presence of an acid catalyst and followed by condensation. The process of obtaining TiO_2 nanoparticles of different shapes and size by the sol-gel method has been studied by Sugimoto et al. (Sugimoto 1987, 1997, 2002a, 2002b, 2003a, 2003b). Eiden-Assmann and co-authors have described a method of obtaining TiO_2 particles of specific target size (Eiden-Assmann 2004). They proposed the use of titanium alkoxide as the initial substance in the alcoholic solution to produce TiO_2 . Unfortunately, this method was proven ineffective as it resulted in often aggregations with a small number of colloidal spherical molecules.

Another method is the non-hydrolytic sol-gel process based on the reaction of titanium tetrachloride with different oxygen-donating compounds such as metal alkoxides or ethers (Niederberger 2002, Parala 2002, Arnal 1996, Burnside 1998). The hydrothermal method has been widely applied to produce particles, mostly of titanium oxides of small diameters for ceramic industry. The nanoparticles of TiO_2 are obtained during hydrothermal treatment by peptisation of titanium precursors with water. The hydrothermal method has many advantages as it needs a lower temperature of crystallisation than in a typical thermal process. The agglomeration of particles can be eliminated or reduced by conducting crystallisation under high pressure. Moreover, high quality products can be obtained without calcination and milling. This method enables control of the shape and size of particles but the process is slow and cannot be implemented on industrial scale (Wu 2002).

The principal users of titania is the paint, varnish, plastic and paper industries. It is also used in production of textiles, pharmaceutical products, cosmetics, cement and

rubber. Recently increasing attention is paid to ultrafine pigments, known as nanopigments, characterised by excellent absorption of ultraviolet radiation and photocatalytic properties. This wide scope of application of this pigment has brought a wide range of commercially available types of titanium dioxide pigments differing in the technology of production, crystalline form, type of surface processing and properties. Increasing demand for titania prompts efforts to improve its quality and to modify certain properties and extension of the gamut of this pigment quality (Braun 1992, Rentschler 1999, Andrzejewska 2004, Jesionowski 2001, 2002a, 2002b, Doerr 1989, Luo 2007, Chen 2003, Hu 1999, Tadros 1996, Tang 2002).

The aim of the study was to obtain titania by precipitation in the emulsion system using titanium(IV) sulphate(VI) as the precursor. The method proposed will permit obtaining pigments based on titanium of anatase-rutile crystallographic structure and hydrophilic/hydrophobic properties.

EXPERIMENTAL

MATERIALS

The Tytanpol[®] R-210 is the rutile variety of the pigment whose surface is processed by aluminium and silicon compounds in the amount of 3% Al_2O_3 and 1% SiO_2 and modified by organic compounds of hydrophilic/hydrophobic nature. The basic parameters describing the properties of this pigment, produced by applying the sulphate method by Chemical Works Police S.A., are given in Table 1.

Table 1. Principal properties of the titanium dioxide R-210

| Physicochemical variable | Titanium dioxide R-210 |
|---|------------------------|
| Content of titanium dioxide (wt. %) | 94 |
| Content of volatile substances at 105°C (wt. %) | 0.5 |
| Content of water soluble materials (wt. %) | 0.5 |
| Residue on a sieve of 45 μm (wt. %) | 0.02 |
| Lightness | 95 |
| Shade in a white paste | -7.0 |
| Relative scattering ability | 100 |
| Ability to tone down the shade | 1850 |
| Shade in a gray paste | 2.5 |
| pH in water suspension | 7.7 |

The substrates used to obtain titania in the emulsion system included: (i) the hydrophobising agent Rokanol K7, oxyethylenated unsaturated fatty alcohol made by

PCC ROKITA S.A.; (ii) cyclohexane (made by POCh S.A.) as the organic phase in the emulsion; (iii) 25% solution of sodium hydroxide (POCh S.A.); (iiii) titanium(IV) sulphate(VI) as the precipitating agent (Chemical Works Police S.A.).

PROCEDURES

Precipitation of titania was carried out in a reactor of 500 cm³ in capacity, in which was placed earlier prepared emulsion of 66 cm³ of cyclohexane, 70 cm³ of a 25% solution of NaOH, Rokanol K7: 3.2–4.4 g. The reagents were vigorously stirred by a homogeniser IKA Werke T25 basic, working at the rate of 13500 rpm. Upon homogenisation the precipitating agent – titanium(IV) sulphate(VI) – was introduced into the reactor at a constant rate of 2.6 cm³/min. by a peristaltic pump ISM833A (Ismatec). Prior to introduction the titanium(IV) sulphate(VI) solution was centrifuged by a high-speed Eppendorf Centrifuge 5804.

The hydrated titanium dioxide was separated from the post-reaction mixture and the salts by filtration under reduced pressure and washed with distilled water on the filter. The product was dried in a stationary dryer at 105°C for about 8h. The dried pigment was calcined in an oven at 920°C for 90 min. In total 7 samples were obtained differing in the amount of the emulsifier used in preparation of the emulsion: sample P1 precipitated from the emulsion containing 3.2 g, P2 – 3.4 g, P3 – 3.6 g, P4 – 3.8 g, P5 – 4.0 g, P6 – 4.2 g and P7 – 4.4 g of Rokanol K7.

PHYSICOCHEMICAL PROPERTIES EVALUATION

Bulk density, absorbing capacity of water, dibutyl phthalate, paraffin oil, oil number and moisture content of the product obtained were measured. The morphology and surface structure of the titanium oxides were analysed by a scanning electron microscope (Zeiss VO40). The particle size and its distribution were measured using the non-invasive backscattering technique (NIBS) on Zetasizer Nano ZS instrument (Malvern Instruments Ltd.). On the basis of the particle size distribution curves the polydispersity was found. The polymorphous variety of the samples was verified by X-ray diffraction on a diffractometer TUR-M62. The colorimetric data of the obtained titanium pigments were documented using an instrumented colorimeter (SPECBOS 4000, JETI Technische Instrumente GmbH), which was calibrated using a white colour standard tile with tristimulus values: X=80.57, Y=84.79, and Z=88.33 (Tytanpol[®] R-210). Daylight (D65) was used as a standardised light source. A fixed amount of pigment sample was poured into the measurement cup. The instrument provided the colour in terms of the CIE $L^*a^*b^*$ colour space system. In this colour space, L^* represented the lightness (or brightness), a^* and b^* were colour coordinates, where $+a^*$ was the red direction, $-a^*$ was the green direction, $+b^*$ was the yellow

direction, and $-b^*$ was the blue direction. Moreover, changes in individual components allowed to estimate the total change of colour ΔE^* . On the other hand, C^* denoted chrome, and h^* determined hue.

RESULTS AND DISCUSSION

The parameters describing the basic physicochemical properties of the TiO₂ powders obtained in the emulsion system are given in Table 2.

Table 2. Principal physicochemical parameters of titania

| Sample name | Emulsifier content (g) | Bulk density (g/dm ³) | Absorbing capacity of (cm ³ /100g) | | | Oil number (cm ³ /100g) | Lightness L* |
|-------------|------------------------|-----------------------------------|---|-------------------|--------------|------------------------------------|--------------|
| | | | water | dibutyl phthalate | paraffin oil | | |
| R-210 | - | 600 | 100 | 95 | 250 | 27 | 93.8 |
| P1 | 3.2 | 660 | 60 | 50 | 160 | 29 | 89.8 |
| P2 | 3.4 | 430 | 95 | 65 | 120 | 28 | 93.3 |
| P3 | 3.6 | 580 | 90 | 60 | 190 | 28 | 89.2 |
| P4 | 3.8 | 540 | 90 | 60 | 185 | 30 | 88.4 |
| P5 | 4.0 | 410 | 90 | 65 | 230 | 30 | 93.4 |
| P6 | 4.2 | 390 | 100 | 70 | 255 | 35 | 93.2 |
| P7 | 4.4 | 430 | 100 | 85 | 260 | 27 | 88.2 |

Analysis of the results has shown that the bulk density of the titania precipitated decreases with increasing content of the emulsifier in the emulsion, even to a value below 400 g/dm³ (the standard sample R-210 density is 600 g/dm³). The absorbing capacities of water, dibutyl phthalate and paraffin oil of the titania precipitated from the emulsion containing 4.4 g of Rokanol K7 (sample P7) are close to the corresponding values of the standard sample of Tytanpol[®] R-210. For both samples, i.e. sample P7 and the standard, the absorbing capacities of water was 100 cm³/100g, of dibutyl phthalate it was 95 cm³/100g for R-210 and 85 cm³/100g for P7 and of paraffin oil it was 250 cm³/100g for R-210 and 260 cm³/100g for P7. The oil number of the sample labelled as P7 is 27 cm³/100g, which is the same as that of the standard R-210. For the other samples obtained the oil numbers are slightly higher. The increasing amount of the surfactant used for the emulsion preparation was found to have insignificant effect on the oil number.

Figure 1 presents graphic illustration of the changes in the colorimetric parameters. The lightness (L*) of the standard sample R-210 is 93.8. The highest value of L* was measured for the samples precipitated from the emulsion containing Rokanol K7 in the amounts of 3.4, 4.0 and 4.2 g, labelled as P2 (L* = 93.3), P5 (L* = 93.4) and P6 (L* = 93.2), respectively. For the other samples L* values were lower and the lowest value

of 88.2 was measured for sample P7. This sample was also found to show the highest hue parameter h^* of 95.5 and the lowest colour saturation parameter (C^*) of 3.5. The lowest h^* of 87.0 was determined for sample P1 precipitated from the emulsion containing 3.2 g of Rokanol K7, while the highest difference in the colour relative to that of the standard

ΔE^* of 9.8 was found for sample P3 precipitated from the emulsion containing 3.6 g of Rokanol K7.

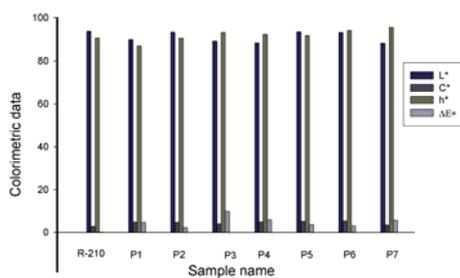


Fig. 1. Colorimetric parameters of titania samples precipitated in the emulsion system

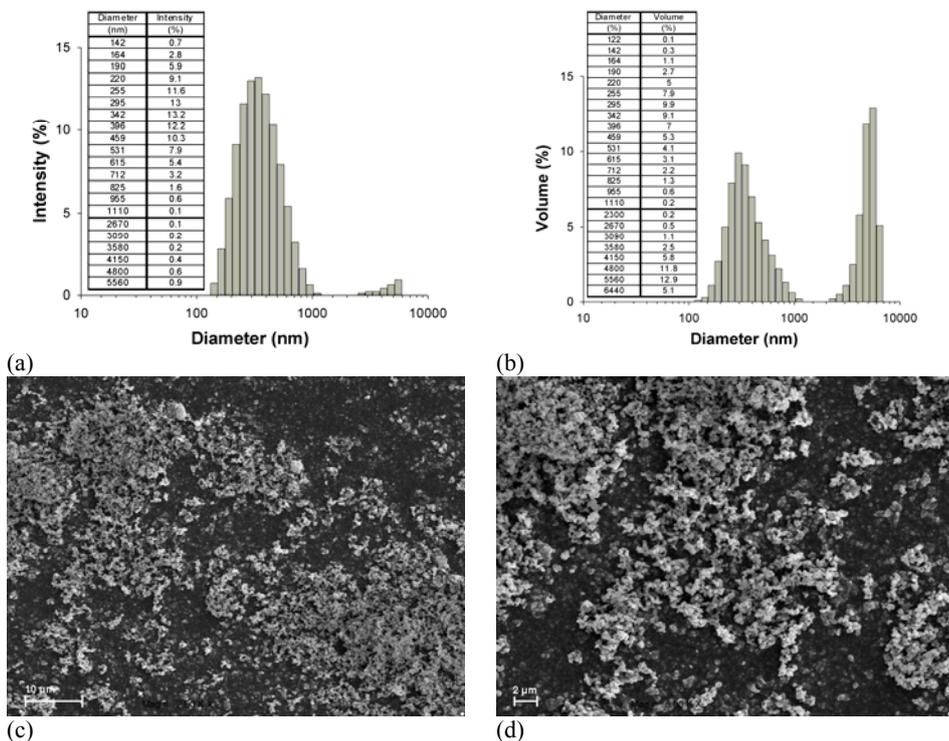


Fig. 2. Particle size distributions: (a) by intensity, (b) by volume and SEM images (c, d) of titanium dioxide R-210 (at different magnifications)

Figure 2 presents the particle size distributions in the form of the percent intensity and volume contribution of particles of a given diameter and the SEM images of the standard sample R-210.

The particle size distribution expressed by the percent intensity contribution of particles of a given diameter (Fig. 2a) shows two bands. The more intense band in the range 142–1110 nm, with the maximum of 13.2% corresponding to the particles of 342 nm in diameter, is assigned to the particles of primary agglomerates. The other band covers the range 2670–5560 nm and is assigned to secondary agglomerates, its maximum intensity of 0.9% corresponds to the particles of 5560 nm in diameter. The polydispersity of R-210 sample related to the particle size distribution is 0.189. The particle size distribution expressed by the percent volume contribution of particles of a given diameter (Fig. 2b) also reveals two bands of similar intensity. The first band in the range 122–1110 nm, with the maximum of 9.9% corresponding to the particles of 295 nm in diameter, is assigned to primary agglomerates. The other band at 2300–6440 nm with the maximum of 12.9% corresponding to the particles of 5560 nm, demonstrates the presence of agglomerates of greater diameters than those of the primary agglomerates. It is concluded from the SEM images, Fig. 2c and d, that the powders are spherical in shape, of small diameters and associate in aggregations and agglomerations.

Figure 3 presents the particle size distributions in the form of the percent intensity and volume contribution of particles of a given diameter and SEM images of sample P1. The particle size distribution expressed as the percent intensity contribution of particles of a given diameter (Fig. 3a) shows two bands. The first more intense band in the range 190–1280 nm, with a maximum of 21.1% for the particles of 396 nm in diameter, is assigned to the primary agglomerates. The other band covering the range 3580–5560 nm, with a maximum of 0.7% corresponding to the particles of 5560 nm in diameter, is assigned to secondary agglomerates. The polydispersity of sample P1 is 0.234, which is higher than that of the standard. The particle size distribution expressed as the percent volume contribution of particles of a given diameter (Fig. 3b) shows two bands. The first at 122–2300 nm, with a maximum of 11.9% for the particles of 396 nm in diameter, is assigned to primary agglomerates. The other band at 4150–6440 nm, with a maximum of 2.6% corresponding to the particles of 4800 nm in diameter, testifies to the presence of secondary agglomerates. The SEM images, shown in Fig. 3c and d, prove the presence of low-diameter particles that are forming aggregations and agglomerations.

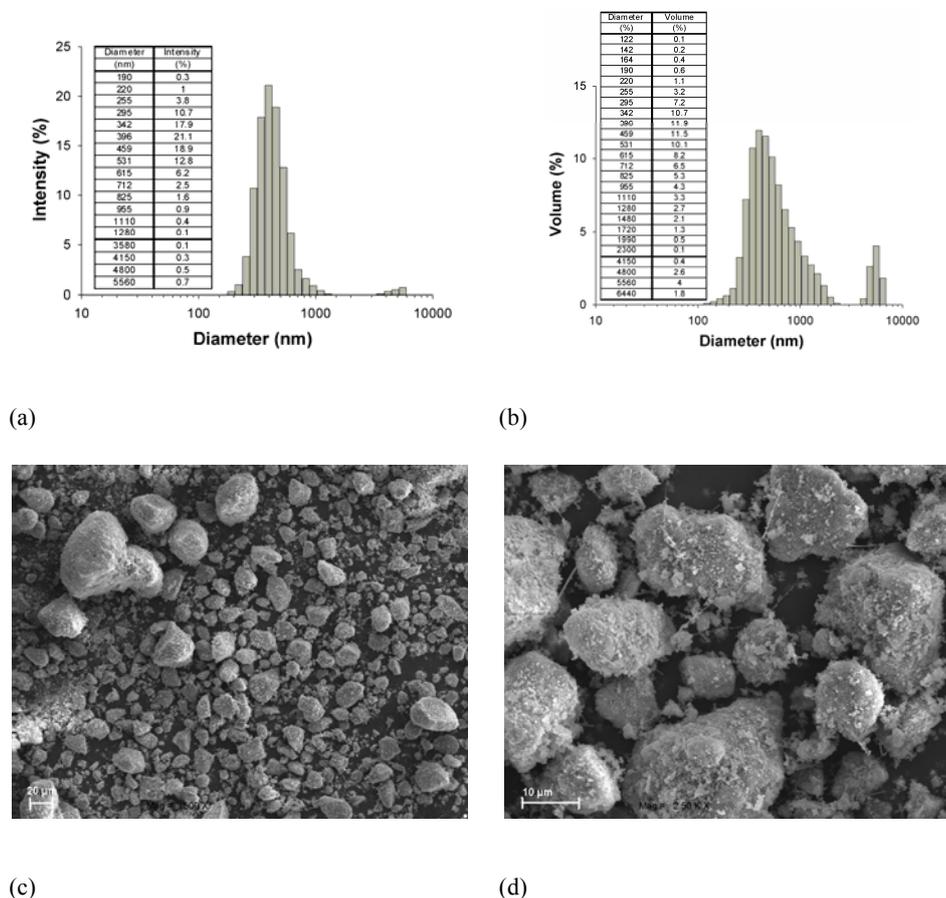


Fig. 3. Particle size distributions: (a) by intensity, (b) by volume and SEM images (c, d) of titanium dioxide precipitated from the emulsion containing 3.2 g of Rokanol K7 (sample P1)

Figure 4 presents the particle size distributions and SEM photographs for sample P3 precipitated from the emulsion containing 3.6 g of Rokanol K7. The particle size distribution expressed by the percent intensity contribution of particles of a given diameter (Fig. 4a) reveals one band only. This band at 220–4150 nm with a maximum of 18.8% corresponding to the particles of 615 nm in diameter, is related to the presence of primary and secondary aggregates. The polydispersity of sample P3 is 0.202. The particle size distribution expressed as the percent volume contribution of particles of a given diameter (Fig. 4b) also shows only one band. This one band covering the range 220–5560 nm, with a maximum of 13.3% for the particles of 615 nm in diameter, is assigned to secondary agglomerates. The SEM images of sample P3, Fig. 4c and d, demonstrate the presence of particles of secondary aggregates associating into agglomerates.

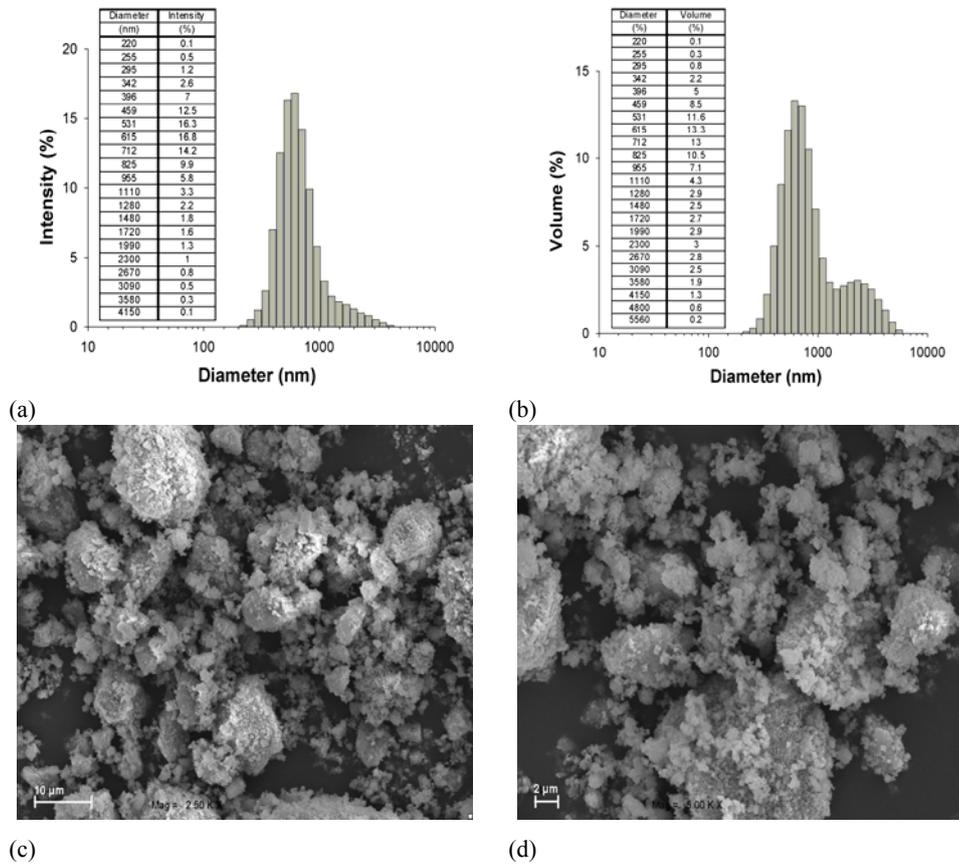


Fig. 4. Particle size distributions: (a) by intensity, (b) by volume and SEM images (c, d) of titanium dioxide precipitated from the emulsion containing 3.6 g of Rokanol K7 (sample P3)

The analogous data and SEM images for sample P5 obtained from the emulsion containing 4.0 g of Rokanol K7 are displayed in Fig. 5.

The particle size distribution expressed by the percent intensity contribution of particles of a given diameter, Fig. 5a, shows one band covering the range 122–5560 nm, with a maximum of 8.3% for the particles of 295 nm in diameter, is assigned to the primary and secondary agglomerates. The particle size distribution expressed by the percent volume contribution of particles of a given diameter, Fig. 5b, also reveals one band in the range 122–6440 nm with a maximum of 6.4% for the agglomerates of 1990 nm indiameter, and is assigned to the primary and secondary agglomerates. The polydispersity of this sample is 0.318, which is greater than that of the above discussed samples (R-210, P1 and P3). The SEM images (Fig. 5c and d) confirm the presence of particles of diameters varying in a wide range.

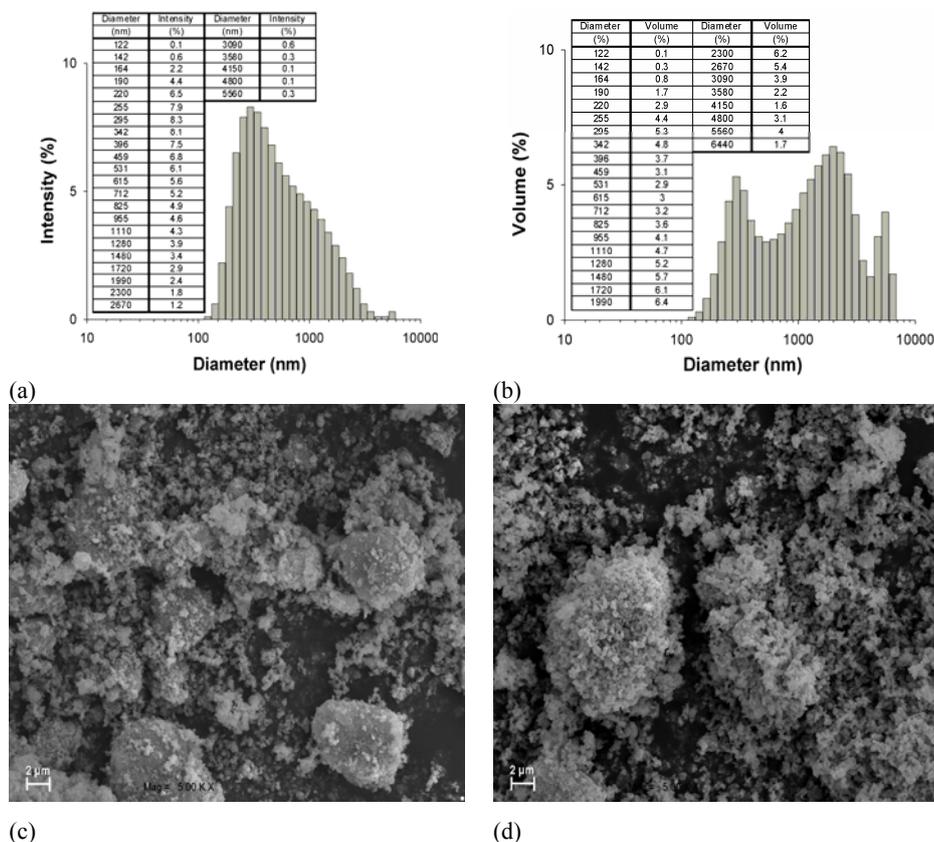


Fig. 5. Particle size distributions: (a) by intensity, (b) by volume and SEM images (c, d) of titania precipitated from the emulsion with 4.0 g of Rokanol K7 (sample P5)

The particle size distributions and SEM images for sample P7 precipitated from the emulsion with 4.4 g of Rokanol K7 are shown in Fig. 6. The particle size distribution expressed by the percent intensity contribution of particles of a given diameter (Fig. 6a) reveals two bands. The first more intense band at 122–1990 nm with a maximum of 14.5% for the particles of 342 in diameter is related to the presence of primary agglomerates. The other band of much lower intensity is assigned to the agglomerates of the diameters from the range 4800–5560 nm. The particle size distribution expressed by the percent volume contribution of particles of a given diameter (Fig. 6b) also shows two bands. The first band, covering the range 122–2300 nm with a maximum of 12.6% for the particles of 342 in diameter, corresponds to the primary agglomerates, while the other, in the range 3580–6440 nm with a maximum of 6.3% for the particles of 5560 nm in diameter, corresponds to secondary agglomerates. The polydispersity of sample P7 is 0.262.

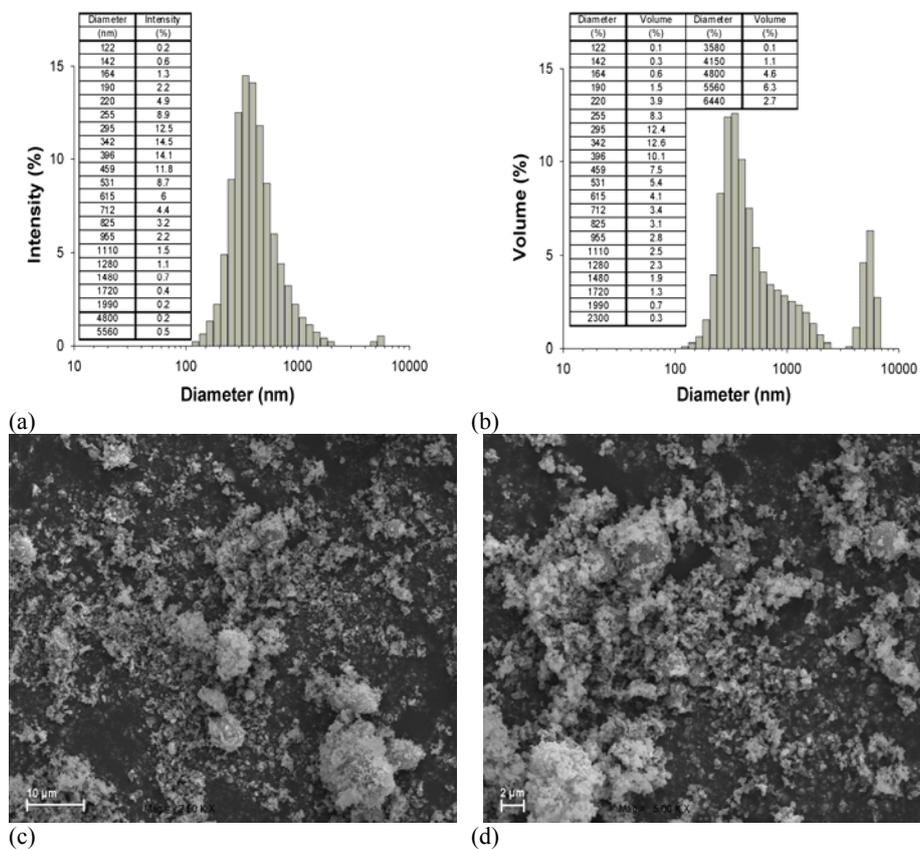


Fig. 6. Particle size distributions: (a) by intensity, (b) by volume and SEM images (c, d) of titania precipitated from the emulsion containing 4.4 g of Rokanol K7 (sample P7)

The crystalline structure of the titania samples obtained by precipitation was determined by X-ray diffraction. Selected diffractograms are shown in Fig. 7. Figure 7a presents the XRD pattern of the standard R-210 sample of rutile type structure, characterised by the maxima for the following values of the angle 2θ: 28, 36, 39, 41, 44, 54, 57. The occurrence of maxima at these values of the angle theta permits identification of the rutile type structure. The X-ray diffraction patterns recorded and shown in Fig. 7b–d confirm the rutile type of the titanium dioxide samples obtained by precipitation, but reveal also samples of anatase type structure, identified on the basis of the maxima for the angle 2θ: 25, 32, 33, 33.5, 48, 54, 55. The X-ray diffractograph of sample P4 shown in Fig. 7c resembles most closely that of the standard sample R-210, which indicates an almost complete transformation of the anatase form into the rutile as a result of calcination of the samples at 920°C.

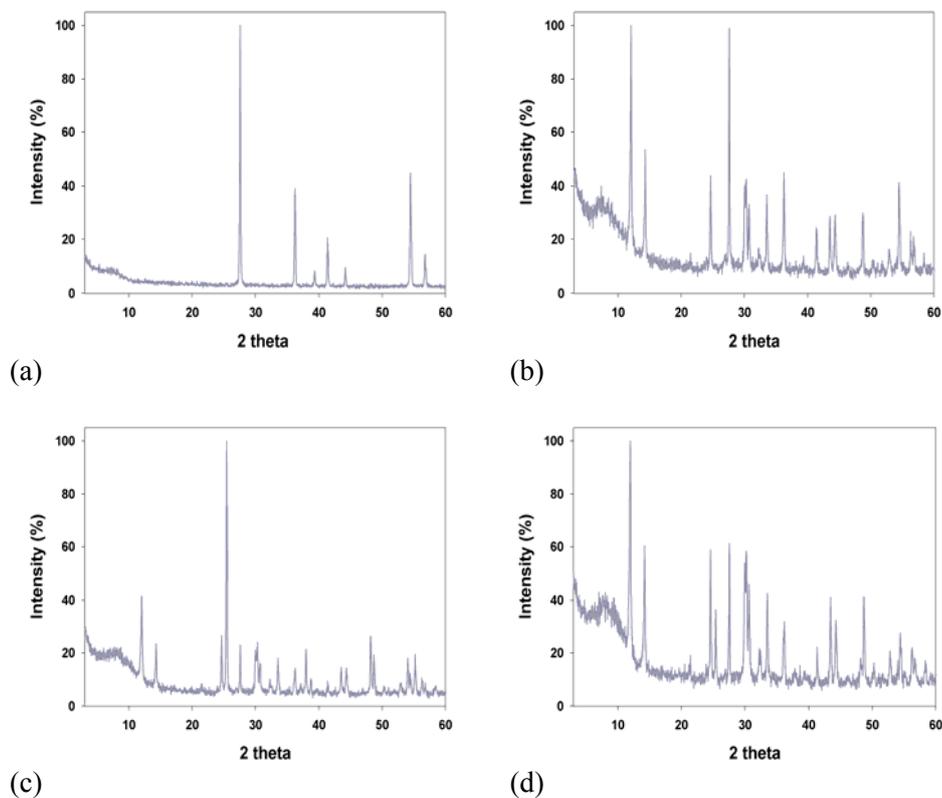


Fig. 7. XRD patterns of selected titanium dioxide samples: (a) R-210, (b) sample P3, (c) sample P4 and (d) sample P7

CONCLUSIONS

The above presented results have confirmed that titania of the rutile type structure can be obtained in the emulsion system. The properties of the TiO_2 samples obtained by precipitation are determined by the emulsifier content in the emulsion. From among all samples obtained the most similar to the standard R-210 was sample P7 precipitated from the emulsion containing 4.4 g of Rokanol K7. This sample contained the primary and secondary agglomerates of the size varying in the range 122–1990 nm, with the dominance of the particles of 342 nm in diameter, similarly as in the standard. The colour of the samples obtained was found to depend significantly on the content of the emulsifier used. The oil numbers of all samples obtained by precipitation are similar.

ACKNOWLEDGEMENTS

This publication was prepared within the key project – POIG no. 01.03.01-00-006/08 cofinanced from the funds of European Regional Development Fund within the framework of the Operational Innovative Economy. The authors thank the Chemical Works Police S.A. for the titanium dioxide standard (TYTANPOL[®] R-210) gift.

REFERENCES

- ANDRZEJEWSKA A., KRYSZTAFKIEWICZ A., JESIONOWSKI T., 2004, *Adsorption of organic dyes on the aminosilane modified TiO₂ surface*, Dyes Pigments, 62, 121-130
- ARNAL P., CORRIU R.J.P., LECLERCQ D., MUTIN P.H., VIOUX A., 1996, *Preparation of anatase, brookite and rutile at low temperature by non-hydrolytic sol-gel methods*, J. Mater. Chem., 6, 1925-1932
- BRAUN J.H., 1997, *Titanium dioxide a review*, J. Coating. Tech., 69, 59-72
- BRAUN J.H., BAIDINS A., MARGANSKI R.F., 1992, *TiO₂ pigment technology: a review*, Prog. Org. Coat., 20, 105-138
- BURNSIDE S.D., SHKLOVER V., BARB C., COMTE P., ARENDSE F., BROOKS K., GRÄTZEL M., 1998, *Self-organization of TiO₂ nanoparticles in thin films*, Chem. Mater., 10, 2419-2425
- BUXBAUM G., 1993, *Industrial Inorganic Pigments*, VCH, Amsterdam
- CHEN Y.-F., LEE C.-Y., YENG M.-Y., CHIU H.-T., 2003, *Preparing titanium oxide with various morphologies*, Mater. Chem. Phys., 81, 39-44
- DĄBROWSKI W., TYMEJCZYK A., LUBKOWSKA A., 2001, *Właściwości i zastosowanie pigmentów dwutlenku tytanu*, Materiały Firmowe Zakładów Chemicznych „POLICE” S.A., Szczecin
- DOERR H., HOLZINGER F., 1989, *Kronos Titandioxid in Dispersionsfarben*, Kronos-Titan GmbH, Leverkusen
- EIDEN-ASSMANN S., WIDONIAK J., MARET G., 2004, *Synthesis and characterization of porous and nonporous monodisperse colloidal TiO₂ particles*, Chem. Mater., 16, 6-11
- HU Z.S., DONG J.X., CHEN G.X., 1999, *Preparation of nanometer titanium oxide with n-butanol supercritical drying*, Powder Technol., 101, 205-210
- JESIONOWSKI T., 2001, *Modification and characterization of titanium dioxide surface*, Pigment Resin Technol., 30, 287-295
- JESIONOWSKI T., KRYSZTAFKIEWICZ A., DEC A., 2002a, *Modified Al₂O₃-treated titanium whites as pigments of acrylic paints*, Physicochem. Problems Mineral. Proc., 36, 307-316
- JESIONOWSKI T., KRYSZTAFKIEWICZ A., DEC A., 2002b, *Modified titanium white covered by Al₂O₃ and SiO₂ - characteristics and application in acrylic paints*, Pigment Resin Technol., 31, 290-296
- KIM K.D., KIM H.T., 2002, *Synthesis of titanium dioxide nanoparticles using a continuous reaction method*, Colloid Surface A, 207, 263-269
- LIU P., BANDARA J., LIN Y., ELGIN D., ALLARD L.F., SUN Y.P., 2002, *Formation of nanocrystalline titanium dioxide in perfluorinated ionomer membrane*, Langmuir, 18, 10398-10401
- LUO Z., CAI H., REN X., LIU J., HONG W., ZHANG P., 2007, *Hydrophilicity of titanium oxide coatings with the addition of silica*, Mat. Sci. Eng. B, 138, 151-156
- MANORAMA S.V., REDDY K.M., REDDY C.V.G., NARAYANAN S., RAJA P.R., CHATTERJI P.R., 2001, *Photostabilization of dye on anatase titania nanoparticles by polymer capping*, J. Phys. Chem. Solids, 63, 135-143
- MORGANS W.M., 1990, *Outlines of Paint Technology*, Halsted Press, New York

- 244 K. Siwińska-Stefańska, A. Krysztafkiewicz, F. Ciesielczyk, D. Pauksza, J. Sójka-Ledakowicz, T. Jesionowski
- NIEDERBERGER M., BARTL M.H., STUCKY G.D., 2002, *Benzyl alcohol and titanium tetrachloride – A versatile reaction system for the nonaqueous and low-temperature preparation of crystalline and luminescent titania nanoparticles*, Chem. Mater., 14, 4364-4370
- PARALA H., DEVI A., BHAKTA R., FISCHER R. A., 2002, *Synthesis of nano-scale TiO₂ particles by a nonhydrolytic approach*, J. Mater. Chem., 12, 1625-1627
- REDDY K.M., REDDY C.V.G., MANORAMA S.V., 2001, *Preparation, characterization, and spectral studies on nanocrystalline anatase TiO₂*, J. Solid State Chem., 158, 180-186
- RENTSCHLER T., RELLER A., 1999, *Optimum particle size is essential*, Eur. Coating. J., 4, 80-89
- SUGIMOTO T., 1987, *Preparation of monodispersed colloidal particles*, Adv. Colloid Interf., 28, 65-108
- SUGIMOTO T., OKADA K., ITOH H., 1997, *Synthesis of uniform spindle-type titania particles by the gel-sol method*, J. Colloid Interf. Sci., 193, 140-143
- SUGIMOTO T., ZHOU X., 2002a, *Synthesis of uniform anatase TiO₂ nanoparticles by the gel-sol method. 2. Adsorption of OH ions to Ti(OH)₄ gel and TiO₂ particles*, J. Colloid Interf. Sci., 252, 347-353
- SUGIMOTO T., ZHOU X., MURAMATSU A., 2002b, *Synthesis of uniform anatase TiO₂ nanoparticles by gel-sol method: 1. Solution chemistry of Ti(OH)_n⁽⁴⁻ⁿ⁾⁺ complexes*, J. Colloid Interf. Sci., 252, 339-346
- SUGIMOTO T., ZHOU X., MURAMATSU A., 2003a, *Synthesis of uniform anatase TiO₂ nanoparticles by gel-sol method: 4. Shape control*, J. Colloid Interf. Sci., 259, 53-61
- SUGIMOTO T., ZHOU X., MURAMATSU A., 2003b, *Synthesis of uniform anatase TiO₂ nanoparticles by gel-sol method: 3. Formation process and size control*, J. Colloid Interf. Sci., 259, 43-52
- TADROS M.E., ADKINS C.L.J., RUSSICK E.M., YOUNGMAN M.P., 1996, *Synthesis of titanium dioxide particles in supercritical CO₂*, J. Supercrit. Fluid., 9, 172-176
- TANG Z., ZHANG J., CHENG Z., ZHANG Z., 2002, *Synthesis of nanosized rutile TiO₂ powder at low temperature*, Mater. Chem. Phys., 77, 314-317
- THE MERCK INDEX, 1996, An Encyclopedia of Chemicals, Drugs and Biologicals, New York
- WU M., LIN G., CHEN D., WANG G., HE D., FENG S., XU R., 2002, *Sol-hydrothermal synthesis and hydrothermally structural evolution of nanocrystal titanium dioxide*, Chem. Mater., 14, 1974-1980
- WYPYCH G., 1999, Handbook of Fillers, 2nd ed., ChemTec Publishing, Toronto

Siwińska-Stefańska K., Krysztafkiewicz A., Ciesielczyk F., Pauksza D., Sójka-Ledakowicz J., Jesionowski T., *Badanie właściwości fizykochemicznych bieli tytanowej otrzymanej w układzie emulsyjnym*, Physicochemical Problems of Mineral Processing, 44 (2010), 231-244, (w jęz. ang), <http://www.minproc.pwr.wroc.pl/journal>

Przeprowadzono badania nad strącaniem bieli tytanowej w układzie emulsyjnym. Wzorcowym ditlenkiem tytanu był Tytanpol® R-210 produkowany przez Zakłady Chemiczne Police S.A. Oznaczano podstawowe parametry fizykochemiczne strąconych próbek ditlenku tytanu, takie jak: chłonności wody, oleju parafinowego i ftalanu dibutyli oraz gęstość nasypową, a także zawartość wilgoci i liczbę olejową. Określano morfologię i mikrostrukturę otrzymanego ditlenku tytanu w celu uzyskania informacji o dyspersji, morfologii ziaren oraz budowie poszczególnych cząstek, wykorzystując techniki SEM i NIBS. Ponadto strącone próbki poddano analizie rentgenograficznej i kolorymetrycznej.

słowa kluczowe: TiO₂, wytrącanie, emulsja, struktura, anataz, rutil, aglomeracja