POSSIBILITIES OF RECOVERING TITANIUM MINERAL FROM MINERAL PIGNEMT BOLUS BY MAGNETIC AND ELECTROSTATIC SEPARATION

MOŽNOSTI ZÍSKÁVÁNÍ MINERÁLŮ Ti Z MINERÁLNÍHO PIGMENTU BOLUS MAGNETICKOU A ELEKTROSTATICKOU SEPARACÍ

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Abstract

This paper deals with the possibilities of using magnetic and electrostatic separation methods for obtaining anatas product from bolus mineral pigment. This pigment, containing, among others, iron and titanium oxides, is an accompanying raw material at brown coal mining, found in the vicinity of excavating premises of the North Bohemian Brown Coal Basin (SHP).

Abstrakt

Příspěvek je věnován možnostem použití magnetických a elektrostatických separačních metod pro získávání anatasového produktu z minerálního pigmentu bolus. Tento pigment, obsahující mimo jiné oxidy železa a titanu, je doprovodnou surovinou při těžbě hnědého uhlí a nachází se v okolí dobývacích prostor Severočeské hnědouhelné pánve (SHP).

Key words: mineral pigments, bolus, anatas, magnetic separation, electrostatic separation

1 INTRODUCTION

The application of natural mineral pigments dates back to the Middle Ages. Since the 1970s, the demand for their production has declined as a result of the advent of organic pigments notable for their colour scheme and affordable price. The increasing requirements for environmental friendliness and chemical stability go together with the effort to replace chemically manufactured pigments with natural materials gradually. On the basis of preliminary research, the properties required for fixing the pigment prices (relevant colour shade, covering power, capacity to absorb oil, high degree of light and thermal stability with minimum physical or chemical changes, low rinse-off of building mixtures, etc.) are favourable for all monitored types of natural pigments based on the raw material resources of the North Bohemian Brown Coal Basin. Properties of these natural mineral colorants can further be enhanced by thermic stabilization, during which more distinctive and steady colour shades are achieved. Today, in addition, demands are raised for low contents of heavy metals as well as environmental friendliness of both the actual raw material and the wastes produced at its processing. In this respect, it would also be suitable to focus particularly on processing wastes from these processes. Thanks to the content of titanium materials in those pigments, it is advisable to try to recover them.

2 RAW MATERIAL CHARACTERISTICS

Highly valued pigments involve the so-called **boluses**, representing montmorillonite or kaolinite clay containing iron oxides, iron hydroxides and titanium. The mineralogical composition of the sample is indicated in Table 1; the granulometric composition in Table 2.

Titanium is the seventh most widespread metal in the earth crust; its content is estimated to be 5.7 – 6.3 g/kg. It occurs in a small amount in most minerals; its most important ores are *ilmenite* - ($FeTiO_3$ ferrous-titanic oxide), *rutile* (TiO_2 – titanic oxide) and *anatas*.[4]

The global titanium production and consumption have remained more or less constant for several years; its slight increase is expected due to the continually widening application possibilities.

Despite its high occurrence in the earth crust, pure metal titanium was a very precious and expensive material for a long time. The reason is that the ordinary metallurgical methods used in the production of other metals are ineffective in the case of titanium owing to its tendency to react with oxygen, hydrogen, carbon and nitrogen at high temperature.

Virtually the most significant titanium compound is *titanic oxide TiO_2*. It is a very stable compound occurring in three modifications in the crystalline state, which correspond to three different minerals – *rutile*, *anatas* and *brookite.*

Anatas may result from chemical processes during solidification of sedimentary rocks. Such anatas crystals are, however, imperceptible, invisible to the naked eye and even under thick magnifying glass. This type occurs in our country in the clays of the North Bohemian Brown Coal Basin and the clays of the Sokolov Brown Coal Basin, where the TiO_2 content amounts even to 8 %, whereas most of the titanium content is made of anatas.

Over 90 % of the world's Ti production is used for the manufacture of TiO_2 pigments (titanium white), as a non-toxic substitute for lead and zinc whites.

The anatas pigment form is primarily used for internal coatings and for the production of paper and plastics. Other application fields include its use as photocatalyst, and particularly its exploitation in the nanotechnologies of surface layer application.

Tab. 1 Mineralogical composition and physical properties of the bolus sample [1]

Mineral name	Chemical composition	Mineral content in the sample [%]	Density [g.cm^{-3}]	Electric conductivity	Magnetic properties
anatas	TiO_2	4,66 ±1,11	3,9	conductive	non-magnetic
hematite	Fe_2O_3	13,77 ± 2,37	5,2	conductive	non-magnetic
kaolinite	$Al_2O_3.2SiO_2.2H_2O$	51,40 ± 5,70	2,6	non-conductive	non-magnetic
quartz	SiO_2	8,42 ± 1,23	2,7	non-conductive	non-magnetic
siderite	$FeCO_3$	21,78 ± 2,55	3,9	non-conductive	magnetic

Tab. 2 Grain-size composition of the bolus sample

Grain-size fraction [mm]	Weight yield [%]	Summary weight yield of oversize [%]	Summary weight yield of undersize [%]
0 – 0,05	19,67	100	19,67
0,05 – 0,08	5,14	80,33	24,81
0,08 – 0,09	1,74	75,19	26,55
0,09 – 0,18	17,22	73,45	43,76
0,18 – 0,25	18,50	56,24	62,27
0,25 – 0,315	21,99	37,73	84,26
0,315 – 0,5	11,30	15,74	95,56
+ 0,5	4,44	4,44	100

3 METHODOLOGY AND PROCEDURES

The executed experimental works were based on the idea of removing the heavily magnetic hematite and siderite minerals by magnetic separation and subsequent separation of non-conductive kaolinite and quartz minerals by electrostatic separation in corona discharge. The final product of the electrostatic separation (conductive product) should be anatas concentrate. Figure 1 shows the flow diagram of the prospective process.

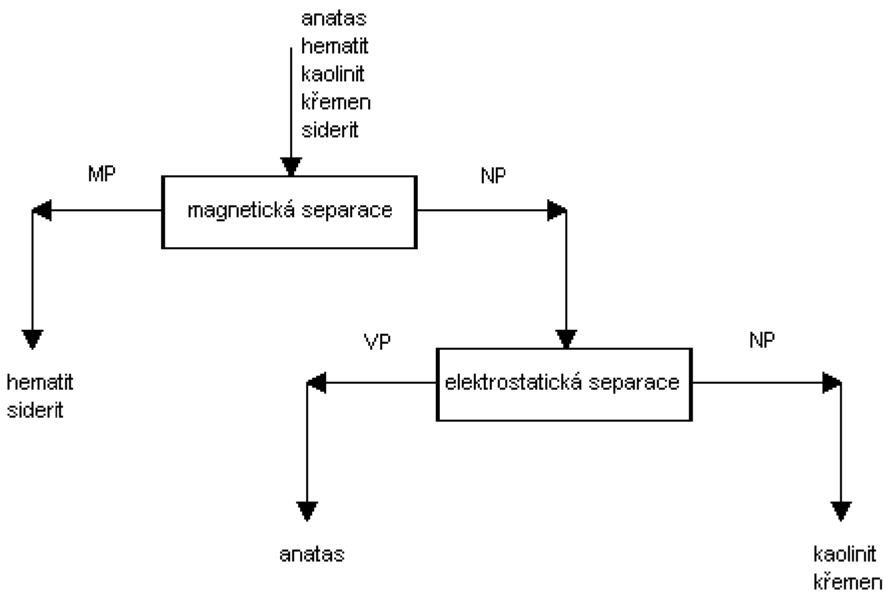

Fig. 1 Flow diagram of the proposed separation process

anatas hematit kaolinit křemen siderit	anatas hematite kaolinite quartz siderite
magnetická separace	magnetic separation
elektrostatická separace	electrostatic separation

To verify the suitability of the proposed procedure, separate tests were executed for dry magnetic separation (induction magnetic separator) and for the wet method (JONES separator – Institute of Geotechnics SAS, Košice). The separation in corona discharge was executed on an electrostatic corona separator at the Institute of Mining Sciences and Environmental Protection of the BERG faculty, TU Košice. To increase the magnetic susceptibility of the sample, magnetic roasting of the sample in a muffle furnace at the temperature of 750 °C was used as well (start-up period: 30 minutes, roasting time: 120 minutes). The material processed like this was again subject to dry magnetic separation.

4 RESULTS AND DISCUSSION

Laboratory experiments focused on the use of magnetic separation were in the first stage executed in a dry way using the magnetic disc separator within the magnetic induction range of 0.15 – 0.83 T. These values of magnetic induction are based on the possibilities of the experimental equipment used. The experiment employed a raw bolus sample of the original grain-size distribution, indicated in Table 2, because further downsizing (grinding) did not seem feasible from technological and economic point of view and the change in the grain-size distribution was not significant during grinding.

It is obvious from the results of the dry magnetic separation (Table 3) that titanium passes uniformly into the individual magnetic products and its behaviour is not influenced by the change in magnetic induction. Apparently, anatas is then very finely intergrown with iron minerals, as evident from the very low titanium content in the non-magnetic product. With view to the fine-grained texture of the raw material, wet magnetic separation was also tested on the polygradient magnetic separator. The results, stated in Table 5, acknowledge the previous statement on the uniform passage of Ti into magnetic products. In term of weight yields, more realistic results were achieved when the larger part of the material passed into the magnetic product only at higher magnetic field induction. Magnetic roasting of the sample caused a substantial increase in the sample's magnetic susceptibility, from the value of 508.72 x 10^{-5} j.SI to 4925,1x10^{-5} j.SI. The results of the subsequent magnetic separation (Table 4) prove a significant change in the material magnetic properties – approx. 70 % of

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the raw material passed into the magnetic product at the lowest induction already. These changes, however, did not manifest itself in any way at the area of Ti passage into the magnetic product.

Tab. 3 Dry magnetic separation

Induction [T]	**Weight yield [%]**	**Ti [%]**	**Fe [%]**	**Ti yield [%]**	**Fe yield [%]**
0.15	3.0	3.51	27.8	3.38	3.49
0.23	12.4	3.38	24.5	13.32	12.60
0.31	27.0	3.34	26.6	28.70	29.83
0.53	43.7	3.15	24.9	43.79	45.17
0.75	9.6	3.07	21	9.41	8.40
0.83	0.6	3.02	10.4	0.59	0.27
NMP	3.6	0.71	1.59	0.81	0.24
Total	**100.0**	**3.15**	**24.10**	**100.00**	**100.0**

Tab. 4 Magnetic separation after roasting

Induction [T]	**Weight yield [%]**	**Ti [%]**	**Fe [%]**	**Ti yield [%]**	**Fe yield [%]**
0.15	73.5	3.76	32.1	75.26	88.31
0.23	10.1	4.65	19.2	12.76	7.24
0.31	3.0	5.05	15.9	4.15	1.80
0.53	3.4	4.17	11.3	3.91	1.45
0.75	3.0	2.68	6.15	2.20	0.69
0.83	2.5	1.34	3.33	0.91	0.31
NMP	4.5	0.66	1.12	0.81	0.19
Total	**100.0**	**3.67**	**26.70**	**100.00**	**100.00**

Tab. 5 Wet magnetic separation

Induction [T]	Weight yield [%]	Volume magnetic susceptibility κ [x 10^{-6} j. SI]	Ti content [%]	Fe content [%]
– 0,15	0,31	10 996,89	3,04	24,7
0,15 – 0,30	0,85	2 468,82	2,68	29,8
0,30 – 0,50	2,45	809,88	2,62	31,8
0,50 – 0,80	15,27	633,73	2,28	32,2
0,80 – 1,20	45,42	561,91	2,46	29,2
+1,20	35,71	230,86	3,91	13,8
Total	**100,00**	**508,72**	2,96	24,2

Tables 6, 7, 8 indicate the results of electrostatic separation in the corona separator.

The sample was first dried up (corona separation is susceptible to material wetness) and then homogenized carefully.

Tab. 6 Conductive product of separation in corona discharge

Revolutions [rpm]	Voltage [kV]	Weight yield [%]	Ti content [%]	Fe content [%]
60	29	57,56	2,58	26,65
60	20	55,30	2,80	20,95
60	10	51,90	2,52	27,90
130	29	62,06	2,37	22,75
130	20	67,72	2,71	26,90
130	10	54,00	2,59	26,25
200	29	72,57	2,58	25,30
200	20	56,27	2,55	27,55
200	10	68,71	2,66	25,3

Tab. 7 Intermediate product from separation in corona discharge

Revolutions [rpm]	Voltage [kV]	Weight yield [%]	Ti content [%]	Fe content [%]
60	29	3,98	2,89	22,85
60	20	4,22	2,54	24,30
60	10	5,36	2,52	24,70
130	29	4,24	2,54	26,55
130	20	9,07	3,05	22,25
130	10	15,70	2,66	24,40
200	29	4,12	2,76	25,15
200	20	17,02	2,71	24,65
200	10	11,80	3,24	20,60

Tab. 8 Non-conductive product from separation in corona discharge

Revolutions [rpm]	Voltage [kV]	Weight yield [%]	Ti content [%]	Fe content [%]
60	29	38,45	3,42	18,50
60	20	40,48	3,53	17,10
60	10	42,74	3,69	18,30
130	29	33,70	3,76	17,55
130	20	22,23	3,57	15,45
130	10	30,31	3,81	16,45
200	29	23,32	4,45	17,00
200	20	26,71	3,79	15,30
200	10	19,50	4,33	16,50

It is obvious from the results of the separation in corona discharge that the separation did not proceed according to the original presumptions (see Figure 1) that anatas would pass into the conductive product. A great deal of TiO_2 passed into the non-conductive product. The fact of very fine mutual intergrowth of the individual raw material components seems to have been proved again.

5 CONCLUSION

The implemented experimental works, focused on the possibility of obtaining anatas product by methods of dry and wet magnetic separation and subsequent electrostatic separation proved that the applied methods seem not to be suitable. Therefore, the required anatas product appears to be unobtainable by classical mechanical processing procedures. In consequence, the following works will be focused on chemical or physicochemical processes of bolus raw material preparation.

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