



Article The Obtaining and Study of Composite Chromium-Containing Pigments from Technogenic Waste

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Abstract: This article provides information on the processing of chromium-containing waste from the Aktobe ferroalloy compounds plant using chemical reagents followed by high-temperature heat treatment for the synthesis of a composite chromite pigment used in the textile industry. This technology was developed for the first time for the purpose of recycling industrial waste and rational use of natural resources. The obtained pigments were analyzed by the X-ray phase of a D878-PC75-17.0 incident beam monochromator and the phase composition of the composite chromite pigment was studied. The thermogravimetric analysis of the composite chromite pigments was performed using a TGA/DSC 1HT/319 analyzer to determine the change in mass with time and temperature. According to the TGA results, the mass loss was determined to be 0.18% of the total mass. The elemental composition of the composite chromite pigment was determined using a JEOL JSM-6490 LV SEM device and the content of chromium oxide (Cr₂O₃) was determined, which reached up to 50%. The thermodynamic patterns of the processes occurring during the production of chromite pigments were studied using the integrated Chemistry software pack HSC-6. The results of testing printed and processed cotton and composite fabrics by the proposed method showed that the color fastness to washing and wet and dry friction is 4 points and the wear resistance assessment is 4860 and 6485 cycles, respectively. Composite chromite pigment based on technogenic wastes is recommended for use in various coloring compositions, including those used for printing on cotton and composite fabrics.

Keywords: synthesis; chromite ores; pigments; environment engineering; industrial waste; recycling

1. Introduction

Today, on the territory of the Republic of Kazakhstan, production sites contain a huge amount of industrial metallurgical waste, which poses a very strong hazard to the environment [1–3]. In recent years, researchers from different countries have been working on the synthesis, characterization and determination of the properties of various types of inorganic pigments obtained from traditional raw materials, as well as by using various industrial wastes from the Aktobe ferroalloy compounds plant in the Republic of Kazakhstan [4–6]. The complete recycling of chrome industrial waste improves the environmental situation and reduces the cost of production of chrome-containing products and pigments [7,8].

Inorganic chromite pigments are widely used in textile production for decorating products, dyeing fabrics and applying patterns to them. In most cases, they are obtained from chemicals under fairly high-temperature treatment [9–11]. This method is expensive, as it requires the purchase of primary raw materials, which in turn increases the cost of the finished products. The technology developed by the authors for obtaining composite chromite pigments based on industrial waste makes it possible to obtain high-quality



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Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). products while simultaneously improving the environmental situation in the region. In pigments, the most common carriers of color are chromophores. These are molecules or atomic groups that have the ability to impart a particular color to the substances in which they are found. The crystal structure of the main phase is often taken into account when classifying pigments. Based on this division, pigments are divided into garnet, spinel, etc. [12,13].

There are also pigments that have a green emerald color and are resistant to high temperatures. These pigments are synthesized on the basis of uvarovite during the high-temperature processing of fluorides and borates. Recently, many researchers have been studying the process of their production [14,15].

The Aktobe ferroalloy compounds plant makes a significant contribution to the contamination with heavy metals of soil in adjacent areas and contributes to the deterioration of the environmental situation in general, since the soil regulates the composition of the atmosphere, hydrosphere and plants growing on it. Due to the fact that soils perform a medical and biological function, which is determined by their impact on human health, their pollution can lead to a number of diseases among the population living in a given area [16,17].

Currently, many countries set technogenic waste utilization, including obtaining valuable components and products from it, as one of their main tasks. The scientific novelty of this work lies in the study of the physical and chemical properties of the chromium oxide in technogenic waste and the development of a technology for obtaining from it a composite chromite pigment.

The aim of this scientific work is the synthesis and study of the properties of inorganic chromite pigments obtained from substandard industrial wastes.

The practical significance of the research work lies in the development of the technological basis for the recycling of technogenic waste through the production of composite chromite pigments, which can replace imported pigments used in the textile industry of Kazakhstan.

The distinctive feature of this technology is based on the synthesis and obtaining of composite chromite pigments from technogenic waste, followed by the application of pigments to cotton and mixed fabric.

The developed composite chromite pigment has a dark green color, imparting color to and increasing the wear resistance of cotton and mixed fabrics.

The technology for obtaining composite chromite pigments from technogenic waste will solve the environmental problem of processing technogenic waste and will substitution the importing of textile pigments. The main consumers of composite chromite pigments are textile industry enterprises, where this product will reduce the cost of finished products.

2. Materials and Methods

2.1. Instrumental Methods of Analysis

In conducting the research work, the authors applied X-ray phase analysis using a D878-PC75-17.0 incident beam monochromator, the Chemistry HSC-6 multifunctional software package, a TGA/DSC 1HT/319 analyzer (Mettler Toledo, Greifensee, Switzerland), a scanning electron microscope (SEM) (JSM-6490IV, Jeol, Tokyo, Japan), an IR Fourier spectrometer (Zhimadzu IR Prestige-21, Tokyo, Japan) and a attenuated total internal reflection device Miracle (Pike Technologies, Kyoto, Japan).

2.2. Pigment Synthesis

A certain mass of substandard composite industrial waste mixture is placed in a planetary mill, crushed and a mass of boric acid is added in a ratio of 1 to 3; the mixture is crushed for 15–20 min until a homogeneous powder is obtained. The crushed mixture is poured into a special container and subjected to temperature treatment from 600 °C to 1000 °C in an oven for 30–45 min. Then, the resulting sinter is cooled and crushed again. The crushed powder is washed several times with water and the resulting suspension

is filtered on a Buchner funnel. The resulting pigment is dried at a temperature of 80 $^\circ\text{C-95}\,^\circ\text{C}.$

The most important moment, on which the reliability of the technology and quality stability of the finished pigment depend, is the method for preparing the batch. The batch prepared according to the recipe was homogenized in a planetary mill by the dry method. The resulting mass was subjected to heat treatment at a temperature range from 600 °C to 1000 °C (in 100 °C increments) with a delay of 2 h at the maximum temperature. The flow chart diagram is shown in Figure 1.



Figure 1. The flow chart diagram of pigment synthesis.

2.3. Thermodynamic Patterns

The chemistry for obtaining chromite pigments based on composite pellets when interacting with a solution of potassium hydroxide:

$$Cr(OH)_3 + KOH \rightarrow KCrO_2 + 2H_2O$$
(1)

$$KCrO_2 + 2H_2O \rightarrow (\gamma - CrOOH) + KOH + H_2O$$
⁽²⁾

Experiments were carried out to determine the ratio of the initial components and thermodynamic parameters using the Chemistry HSC-6 software pack again, in a temperature range of 0–1000 °C with an increase in temperature in intervals of 100 °C for the formation of chromite pigment processed with alkaline solutions to obtain potassium chromite (Table 1) and the subsequent production of chromite pigments (Table 2).

Table 1. Chemical parameters of starting and final substances for reaction 1.

Formula	FM, g/mol	Conc.wt-%	Amount, mol	Amount, g	Volume, mL
Cr(OH) ₃	103.018	64.741	1.000	103.01	0.000
КОН	56.106	35.259	1.000	56.106	27.449
KCrO ₂	123.093	77.357	1.000	123.093	0.000
H ₂ O	18.015	22.643	2.000	36.030	39.292

Table 2. Chemical parameters of starting and final substances for reaction 2.

Formula	FM, g/mol	Conc.wt-%	Amount, mol	Amount, g	Volume, mL
KCrO ₂	123.093	77.357	1.000	123.09	0.000
H ₂ O	18.015	22.643	2.000	36.030	39.292
CrO(OH)	85.003	53.419	1.000	85.003	22.414
КОН	56.106	35.259	1.000	56.106	27.449
H ₂ O	18.015	11.322	1.000	18.015	19.646

3. Results and Discussion

The closest to our technology, in terms of technical essence and the achieved result, is the method of obtaining emerald chrome pigment, which includes calcining the batch containing a chromium compound, boric acid and carbamide, followed by washing and drying the pigment [18].

This technology is ineffective because it has a complex technological process. Also, the product obtained by this technology does not reach the color required to be considered emerald.

The difference in the technology proposed by the authors is in the improvement of the technology for obtaining composite chromite pigments by optimizing the technological process and eliminating the use of autoclave equipment. In addition, in the technology developed by the authors, it is proposed to use industrial wastes as raw materials.

3.1. Results of X-Ray Phase Analysis

The structural state and chemical phase composition of the composite chromite pigment have been determined, the results of which are shown in Figure 2.



Figure 2. X-ray phase analysis of chromite pigments.

The presence of the following phases in the pigments was confirmed by X-ray phase analysis: $Ca_3Fe_2Si_3O_{12}$ —andradite, Ca_3iO_3 —wollastonite, Fe_2O_3 —hematite, SiO_2 —cristobalite, $Ca_3Cr_2Si_3O_{12}$ —uvarovite, Cr_2O_3 —chromium oxide and SiO_2 —quartz. With increasing temperature, an increase in the intensity of the peaks of the main phases—andradite and uvarovite—is noted [19–22].

3.2. Results of Thermogravimetric Analysis

To study the change in the chromite pigments mass depending on time and temperature, a thermogravimetric analysis of the chromite pigments was carried out using a TGA/DSC 1HT/319 analyzer (Mettler Toledo, Greifensee, Switzerland). The results of the analysis are shown in Figure 3.



Figure 3. Thermogravimetric analysis of chromite pigments.

From Figure 3, the mass of the chromite pigments depends on time and temperature. This is evidenced by the changes in their mass at different temperatures: (1) 200.19 °C— 100.299 mg; (2) 380.33 °C—100.218 mg; (3) 510.48 °C—100.211 mg; (4) 674.05 °C—100.166 mg; (5) 933.03 °C —100.200 mg; (6) 990.21 °C—100.182 mg. According to the results of the thermogravimetric analysis, it was found that the chromite pigment mass loss in the temperature range of 200.19–990.18 °C within 94 min is 0.117 mg.

3.3. Results of Thermodynamic Study

As a result of thermodynamic studies of the two reactions, the values of enthalpy (Δ H), entropy (Δ S) and Gibbs energy (Δ G) in a heterogeneous system were determined using the complex Chemistry HSC-6 program. The results of the thermodynamic studies are given in Table 3.

					Gibbs E	energy ΔG,	, kJ/mole				
Reaction No.					Te	mperature,	, °C				
	0	100	200	300	400	500	600	700	800	900	1000
(1)	-68.7	-71.8	-88.5	-104.2	-123.5	-145.7	-171.1	-199.1	-230.9	-264.7	-300.8
(2)	-188.6	-190.2	-193.6	-195.8	-198.2	-201.4	-207.7	-212.8	-217.4	-224.3	-228.6

Table 3. Change in the Gibbs energy for reactions 1 and 2.

From Table 3, it follows that with an increase in a temperature from 0 to 1000 $^{\circ}$ C, the Gibbs energy for reaction (1) increases from -68.7 to -300.8 kJ/mole and becomes negative, and the possibility of this chemical reaction is explained by the negative value

of the Gibbs energy. In the second reaction, reaction 2, with an increase in temperature to 1000 $^{\circ}$ C, the Gibbs energy value slowly increases from -188.6 to -228.6 kJ/mole.

3.4. Results of IR Spectrum and Elemental Analysis

To determine the functional groups in the chromite pigments, spectral studies were carried out using an IR spectrometer (Shimadzu IR Prestige-21). The results of the studies are shown in Table 4 and Figure 4.

No	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	690.52	80.184	5.767	721.38	617.22	8.504	2.074
2	763.81	85.108	0.224	771.53	744.52	1.855	0.014
3	779.24	83.190	2.559	856.39	771.53	4.460	0.143
4	918.12	84.117	8.178	968.27	867.97	5.055	1.542
5	1022.27	86.445	6.458	1060.85	972.12	3.909	1.099
6	1095.57	88.145	5.404	1149.57	1064.71	3.230	0.899
7	1192.01	91.113	2.819	1207.44	1153.43	1.573	0.235
8	1249.87	88.846	2.526	1265.30	1211.30	2.237	0.309
9	1334.74	83.347	4.948	1388.75	1269.16	7.946	1.623
10	1419.61	86.261	1.587	1550.77	1404.18	5.301	0.559
11	1651.07	91.501	1.900	1678.07	1581.63	0.542	0.359
12	2260.57	101.108	0.491	2283.72	2214.28	-0.426	0.054
13	2376.30	101.396	0.298	2403.30	2364.73	-0.270	0.019
14	3197.98	92.823	2.974	3294.42	2916.37	7.256	2.604
15	3363.86	93.606	1.228	3421.72	3321.42	2.549	0.270

Table 4. Spectral data of chromite pigments.





Figure 4 shows the IR spectrum of the chromite pigments, from which it follows that: absorption spectra with wavelengths of 3363.86–3197.98 cm⁻¹ characterize the presence of hydroxyl groups (CrO-OH); absorption spectra with wavelengths of 2376.30–2260.57 cm⁻¹ characterize the presence of chromite pigments groups (KCrO₂); absorption spectra with

wavelengths of 1651.07 cm⁻¹ characterize the presence of chromite pigments groups (K₂CrO₄); absorption spectra with wavelengths in the range of 1419.61 cm⁻¹ characterize the presence of chromite pigments groups (K₃CrO₄); absorption spectra with wavelengths in the range of 1334.74 cm⁻¹ characterize the presence of chromite pigments groups (K₄CrO₄); absorption spectra with wavelengths in the range of 1249.87–1192.01 cm⁻¹ characterize the presence of chromite pigments groups (K₂O·Cr₂O₆); absorption spectra with wavelengths in the range of 1095.57–1022.27 cm⁻¹ characterize the presence of chromite pigments groups (K₂O·Cr₂O₆); absorption spectra with wavelengths in the range of 918.12–779.24 cm⁻¹ characterize the presence of chromite pigments groups (CrO₂); absorption spectra with wavelengths in the range of 763.81–690.52 cm⁻¹ characterize the presence of chromite pigments groups (CrO₂).

The elemental composition and micrographs of the composite chromite pigment are shown in Figure 5.





From Figure 5 it follows that in the composition of chromite pigment, chromium oxide (Cr_2O_3) reaches a content of up to 50%. This content of a valuable component is enough to use chromite pigment as dyes in the textile industry.

3.5. Application of Pigment to Fabric and Its Durability

Chromite pigments obtained from technogenic wastes from the Aktobe ferroalloy compounds plant, dispersants and auxiliary materials from the company "Archroma" (Switzerland), were used as the initial components for the dyes. The recipe for the printing paste used during the tests is given in Table 5.

The printing machine printed test strips 4 cm wide on a cotton fabric Alatau article 1001 (175 g/m²) and a composite (PES 53%, cotton 47%) fabric Arlan article 6005 (175 g/m²) in the amount of 25 strips of each option. The cotton fabric Alatau article 1001 (175 g/m²) is used as the main fabric for a summer camouflage jacket with straight-cut trousers. Warp threads—100% cotton—Nm34 (29.4 Tex), fabric density by warp is 28 threads/cm; weft threads—100% cotton—Nm20 (50.0 Tex), fabric density by weft is 19 threads/cm.

The composite fabric (PES 53%, cotton 47%) Arlan article 6005 (175 g/m²) is used as the main fabric for a summer field camouflage jacket with straight-cut trousers. Warp threads—100% polyester—Nm34 (29.4 Tex), fabric density by warp is 36 threads/cm; weft threads—100% cotton–Nm27 (37.04 Tex), fabric density by weft is 21 threads/cm. The dyeing strength tests were carried out according to GOST 9733.0-83 "Textiles. General requirements for test methods of color fastness to physical and chemical actions" [23–25].

Studies of the color characteristics of pigments from technogenic wastes applied to different types of fabrics showed that when using the same paste recipe, the Alatau fabric, article 1001, was dyed SW6473 Surf Green, and the Arlan blended fabric, article 6005, was dyed SW6474 Raging Sea. The results of the studies are shown in Figure 6a,b, respectively.

Chemicals for Mother Thickener	UM	Mother Thickener	Thickener for Dilution	
Softened water	(g/kg)	687	948	
Antimussol [®] UDF Liq (1:2 in water)	(g/kg)	1	1	
Ammonia (25%)	(g/kg)	2	2	
Urea	(g/kg)	20	20	
Printofix [®] Softener	(g/kg)	10	5	
Printofix [®] Fixer	(g/kg)	10	5	
Printofix [®] Binder	(g/kg)	250	0	
Printofix [®] Thickener	(g/kg)	20	19	
Total	(g)	1000	1000	
Printing paste				
Mother thickener	(g/kg)	Х	424	
Thickener for dilution	(g/kg)	Y	560	
Chromite pigment	(g/kg)	Z	16	

Table 5. The recipe of the printing paste used during the tests.



Figure 6. Samples of fabrics with applied pigment from technogenic wastes. (**a**) Cotton fabric Alatau article 1001. (**b**) Composite fabric Arlan article 6005.

4. Conclusions

A chromite pigment was synthesized based on chromium-containing waste from the Aktobe ferroalloy compounds plant using chemical reagents. An X-ray phase analysis was performed using a D878-PC75-17.0 device and the phase composition of the composite chromite pigment was studied. Thermogravimetric analysis was performed to determine the change in mass over time and temperature. According to the TGA results, it was determined that the mass loss was 0.18% of the total mass.

The thermodynamic patterns of the ongoing chemical processes during the production of chromite pigments were studied using the integrated Chemistry HSC-6 program. The thermodynamic parameters of the heterogeneous system were determined; namely the values of enthalpy Δ H, entropy Δ S and Gibbs energy Δ G. A negative Gibbs energy value confirms the occurrence of chemical processes during the production of chromite pigments.

IR spectral analysis was performed using an IR spectrometer (Shimadzu IR Prestige-21) to determine the inorganic functional groups present in the composite chromite pigments. The elemental composition of the composite chromite pigment was also determined using scanning microscopy (JSM-6490IV, Jeol, Tokyo, Japan). According to the results of the elemental analysis, the content of chromium oxide (Cr_2O_3) reached up to 50%. This content of chromium oxide in the composite chromite pigment is sufficient for its use as a dye in the textile industry.

According to the results of photocolorimetric analysis, it was determined that the final color of the fabric is affected not only by the recipe of the printing paste, but also by the fibrous composition of the fabric sample used. Samples of fabric with a printed pattern were subjected to physical mechanical and physical chemical tests in accordance with the requirements of the technical regulations of the Customs Union TR CU 017/2011 "On the safety of light industry products". The results of testing printed and processed cotton and composite fabrics according to the proposed method showed that the color fastness to washing and wet and dry friction is 4 points, and the wear resistance assessment is 4860 and 6485 cycles, respectively.

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