Environmental Science and Pollution Research (2021) 28:32660–32668
https://doi.org/10.1007/s11356-021-13007-4

RESEARCH ARTICLE

Pigments from spent Zn, Ni, Cu, and Cd electrolytes from electroplating industry

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Received: 16 November 2020 / Accepted: 12 February 2021 / Published online: 24 February 2021

Abstract

One of the problems of electroplating industry is the periodic discharge of concentrated spent electrolytes together with rinsing wastewater. This leads to irreversible loss of valuable components, as well as to the risk of heavy metal ions entering the environment, which have toxic, mutagenic, and carcinogenic effects. The paper presents research on the processing of spent electrolytes from electroplating industry of zinc, nickel, copper, and cadmium plating, collected over 3 years. Pigments of various colors were obtained by precipitation of zinc, nickel, copper, and cadmium ions by phosphate, hydroxide, and sodium carbonate. By their properties, i.e., whiteness 95–97%, residue after sieving on a sieve up to 0.04 wt.%, etc., the resulting pigments are not inferior to those currently presented on the world market. Following previous studies, a basic technological scheme for processing waste electrolytes with pigments production is proposed. Processing of spent electrolytes according to the proposed technology will make it possible to reduce the concentration of heavy metal ions to acceptable values (0.13–0.65 mg/L) for discharge. This will ensure stable and uninterrupted operation of local treatment facilities of electroplating industry.

Keywords Electroplating industry · Waste recycling · Spent electrolyte · Pigment

Introduction

At present, galvanic coatings are widely used in mechanical engineering to protect products from corrosion and impart necessary properties (increased wear resistance, hardness, etc.). However, the technology of their application involves formation of, i.e., sewage sludge, galvanic sludge, technological waste solutions. The most dangerous waste is spent electrolytes since they have high content of heavy metal ions, which produce many toxic (carcinogenic, mutagenic, etc.) effects (Martsul et al. 2012a, b).

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The recycling of industrial wastes with the production of valuable materials is of interest (Romanovski 2021; Romanovskaia et al. 2021; Romanovski 2020; Kamarou et al. 2020). Different spent materials were considered as a source for pigment production, for example, spent batteries as raw materials of pigments: spent zinc-carbon batteries (Rodrigues et al. 2018), spent alkaline batteries (Almeida et al. 2020), and some others like Al-rich sludge (generated in the wastewater treatment unit of an anodizing or surface coating industrial plant), a galvanizing sludge (from the Cr-/Ni-plating process), and Fe-rich sludge (generated during steel wiredrawing) (Hajjaji et al. 2010; Carneiro et al. 2019). Galvanic sludge is formed during the treatment of rinsing wastewater from electroplating industry. Their composition depends on the cleaning method (reagent, electrocoagulation, galvanocoagulation, etc.). Most often, it is a mixture of hydroxides, carbonates, less often sulfides of heavy metals (zinc, nickel, chromium, copper, cadmium, lead, etc.), calcium, and magnesium, as well as ferrous compounds (Chiu et al. 1987). Besides, galvanic sludge accumulates directly in the electroplating baths due to the formation of insoluble impurities during the electrolyte operation. After dehydration, sewage sludge and galvanic sludge are stored in special metal containers, sedimentation tanks, or sludge collectors. The

waste occupies useful areas and causes the risk of heavy metal ions entering the environment. A number of studies are devoted to the processing of sewage sludge and galvanic sludge to obtain metals for metallurgy. These include chromium (Silva et al. 2006); separation of chromium, nickel, iron, zinc, and copper salts (Li et al. 2010); copper and nickel (Veglio et al. 2003); copper (Jandova et al. 2000); copper, nickel, and zinc (Amaral et al. 2014); and copper, zinc, and nickel (Rossini and Bernardes 2006).

There are not many studies that describe production of commercial goods (Carneiro et al. 2018). Articles (Costa et al. 2008; Ferreira et al. 1999; Abdurakhmanov et al. 2000) present the results of using galvanic sludge in the production of ceramic products for various purposes such as clay bricks (Pérez-Villarejo et al. 2015), cement clinker (Espinosa and Tenório 2000; Ract et al. 2003), different glasses (Andreola et al. 2006; Silva et al. 2008), and obtaining pigments (Cheprasova and Zalyhina 2017; Andreola et al. 2008).

Not enough attention is paid to the recycling of spent electrolytes which are in most cases regenerated. The number of regeneration cycles is important but is limited by the accumulation of impurities, which are not possible to remove. Therefore, it becomes necessary to replace spent electrolytes, which can also occur due to a change in their composition or the type of manufactured products.

According to our studies (Martsul et al. 2013; Martsul et al. 2012a, b) carried out over 3 years at more than 30 enterprises of the Republic of Belarus (the country is located in the central part of the European continent), spent electrolytes are often considered as wastewater and discharged to treatment facilities along with rinsing wastewater where the concentration of heavy metal ions is hundreds of times lower compared with the first ones. This increases the load on treatment facilities and, accordingly, reduces the efficiency of wastewater treatment or requires a significant amount of clean water for preliminary dilution of concentrated spent electrolytes. However, spent electrolytes are industrial waste, which raises the question of the legality of their discharge to treatment facilities. Therefore, it is advisable to dispose of rinsing wastewater and spent electrolytes separately with subsequent processing of the electrolytes. The high concentration of heavy metal ions in the spent electrolytes, the compounds of which have chromophore properties, makes them promising for the production of pigments. Currently, there is no production of pigments in the Republic of Belarus, and they are purchased from abroad (China, Germany, Spain, Czech Republic, etc.). It should also be noted that in the Republic of Belarus there are no resources for their production.

The range of electroplated coatings depends on their intended use. In general, according to the types of coatings, the volume of their production in the world is distributed in percentage as follows: zinc plating 40–50%, nickel plating 8–10%, copper plating 7–8%, chrome plating 7–8%, cadmium plating 4–5%, others 18–33% (Costa et al. 2008).

The objectives of the study are as follows: (i) to isolate heavy metal ions from the spent electrolytes of galvanic production in the form of a precipitate; (ii) to determine the mode of heat treatment of Zn-, Ni-, Cu-, Cr-, and Cd-containing sediments obtained during deposition from spent electrolytes of electroplating industry; (iii) to determine the composition, structure, and properties of the obtained compounds to confirm the possibility of their use as pigments; (iv) to offer a technological scheme for obtaining pigments from spent Zn, Ni, Cu, Cr, and Cd electrolytes of electroplating industry.

Materials and methods

Materials and reagents

Spent electrolytes from galvanizing, nickel plating, copper plating, and cadmium plating from various Belarusian enterprises were selected for research. Sampling of the spent electrolytes was carried out for 3 years. The studies were carried out on 24 samples of spent galvanizing electrolytes, 18 samples of spent nickel-plating electrolytes, 15 samples of spent copper-plating electrolytes, and 9 samples of spent cadmium-plating electrolytes.

The main components of the studied spent galvanizing electrolyte are zinc chloride $ZnCl_2$ (37–109 g/L) and ammonium chloride NH_4Cl (133–277 g/L). The composition of the spent nickel-plating electrolyte selected for research included nickel sulfate $NiSO_4$ (132–277 g/L), boric acid H_3BO_3 (30–50 g/L), and sodium chloride NaCl (10–25 g/L). The spent copper-plating electrolyte contained copper sulfate $CuSO_4$ (77–104 g/L) and sulfuric acid H_2SO_4 (30–50 g/L) as the main components. The spent cadmium-plating electrolyte contained cadmium sulfate $CdSO_4$ (34–56 g/L) and sulfuric acid H_2SO_4 (25–30 g/L) as the main components. In addition to the main components, the composition of spent electrolytes includes various impurities in micro amounts: iron ions, brightening additives, wetting agents, leveling additives, etc.

Synthesis

Zinc-containing pigments were obtained by precipitation of Zn(II) ions with saturated (at 25 °C) solutions of sodium carbonate and phosphate, nickel-containing pigments—by precipitation of Ni(II) ions with saturated solutions of sodium hydroxide and phosphate, copper-containing ones—by precipitation of Cu(II) ions with a saturated solution of sodium phosphate, cadmium-containing—by precipitation of Cd(II) ions with a saturated solution of sodium carbonate. The resulting precipitates were aged under a layer of mother liquor

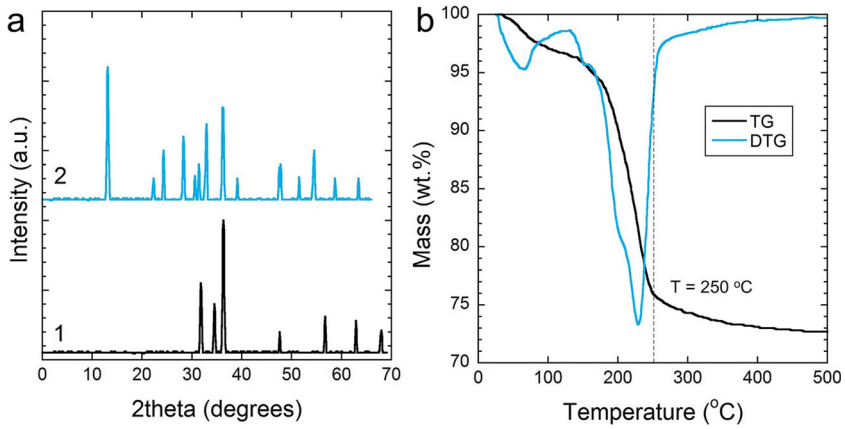

Fig. 1 **a** XRD patterns: 1—after drying; 2—after heat treatment and **b** the thermogravimetric analysis of the precipitate obtained by the precipitation of zinc ions from the spent zincing electrolyte with sodium carbonate solution

for 30 min, separated from the solution, washed from water-soluble salts, and dried at 80 °C for 3 h. The temperature for heat treatment was determined by the data of thermogravimetric analysis.

Material characterization

The concentration of zinc, nickel, copper, and cadmium ions in the spent electrolytes was determined by titrimetric method according to (Rice 2012).

X-ray phase analysis of the obtained samples was carried out on a D8 Advance Bruker AXS X-ray diffractometer (Germany). The recording was carried out in the range of angles 2θ 10–80° with a step of 0.1–0.2° and the accumulation of pulses for 2 s. The obtained XRD patterns were identified using the Match1.10.1.446 software and the ICDD PDF-2 database.

The elemental composition of the obtained sediments and the study of their microstructure were carried out on a JSM-5610 LV scanning electron microscope with an EDXJED-2201 chemical analysis system (JEOL, Japan).

Thermogravimetric analysis was done using a TGA/DSC-1/1600 HF thermoanalytical system (METTLER TOLEDO Instruments, Switzerland) using Al_2O_3 as a standard (platinum crucibles, heating rate 10 °C/min). On the basis of a joint analysis of the TG, DTG, and DTA curves, the heat treatment modes were justified.

The oil absorption was determined according to (ISO 787-9 2017) and its analog GOST 21119.8-75. The method consists in determining the amount of linseed oil, which is necessary to form a flowable paste pigment (oil absorption I kind) and no crumbling lump (oil absorption kind II).

A three-dimensional model of the CIE 1976 $L^*a^*b^*$ color space was used to characterize the color of the obtained samples. In this model, the color was determined by the luminance L^* and the two chromatic components a^* and b^*, which correspond to a color change from green to red and from blue to yellow, respectively. The L^* brightness ranges from 0 to 100 (darkest to lightest). The color characteristics of the obtained samples were determined on an autonomous universal spectrophotometer ColorEyeXTH (GretagMacbeth).

The pH of the aqueous suspension of the pigment was determined according to (ISO 787-9 2019). The sieve residue was determined according to (EN ISO 787-18 2019).

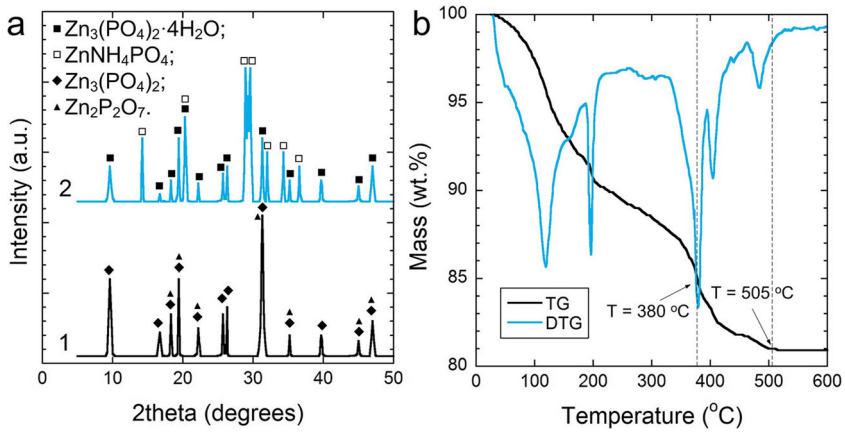

Fig. 2 **a** XRD patterns: 1—after heat treatment; 2—after drying and **b** the thermogravimetric analysis of the precipitate obtained by the precipitation of zinc ions from the spent zincing electrolyte with sodium phosphate solution

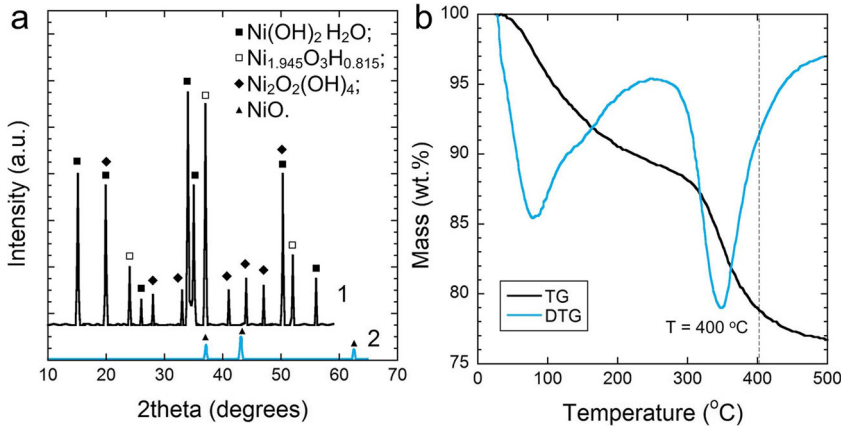

Fig. 3 **a** XRD patterns: 1—after drying; 2—after heat treatment and **b** the thermogravimetric analysis of the precipitate obtained by the precipitation of nickel ions from the spent nickel-plating electrolyte with sodium hydroxide solution

Results and discussion

Pigment synthesis

Reagents for the deposition of zinc, nickel, copper, and cadmium ions were chosen taking into account the possibility of obtaining compounds that are currently used as pigments. The chemical composition and color characteristics of known zinc-, nickel-, copper-, and cadmium-containing pigments are presented in Table S.1.

Synthesis of Zn-based pigments

Based on the composition of the spent electrolytes and the composition of currently used pigments (Table S.1), sodium carbonate and phosphate were chosen as precipitants for zinc ions, sodium hydroxide and phosphate for nickel ions, sodium phosphate for copper ions, and sodium phosphate for cadmium ions.

During the precipitation of zinc ions with sodium carbonate, zinc hydroxycarbonate (card #01-072-1100, $Zn_5(OH)_6(CO_3)_2$) was formed, which was confirmed by the data of X-ray phase analysis (Fig. 1a, pattern 1).

Heat treatment temperature of the obtained sample was established according to the data of thermogravimetric analysis (Fig. 1b) and amounted to 250 °C. This was accompanied by the formation of white zinc oxide (Fig. 1a, pattern 2) and corresponds to card #00-036-1451.

According to X-ray phase analysis data, zinc phosphate tetrahydrate $Zn_3(PO_4)_2 \cdot 4H_2O$ and zinc-ammonium phosphate $ZnNH_4PO_4$ (cards #00-037-0465 and #00-022-0025 respectively) were formed during the deposition of zinc ions with sodium phosphate (Fig. 2a, pattern 2) due to the high content of ammonium chloride in the spent electrolyte. Based on thermogravimetric analysis (Fig. 2b), a two-stage heat treatment mode was selected with hold down at temperatures of 380 and 505 °C for an hour. The phase composition of the sample after firing is represented by a mixture of phosphate $Zn_3(PO_4)_2$ and zinc diphosphate $Zn_2P_2O_7$ (Fig. 2a, pattern 1), cards #00-029-1390 and #00-049-1240 respectively.

Synthesis of Ni-based pigments

When nickel ions were precipitated with sodium hydroxide, a mixture of nickel hydroxides of non-stoichiometric composition was formed $Ni(OH)_2 \cdot 2H_2O$, $Ni_{1,945}O_3H_{0,815}$, and $Ni_2O_2(OH)_4$, cards #00-022-0444, #01-084-1176, and #00-013-0229 (Fig. 3a, pattern 1).

Based on thermogravimetric analysis (Fig. 3b), the heat treatment temperature of the obtained precipitate was set at

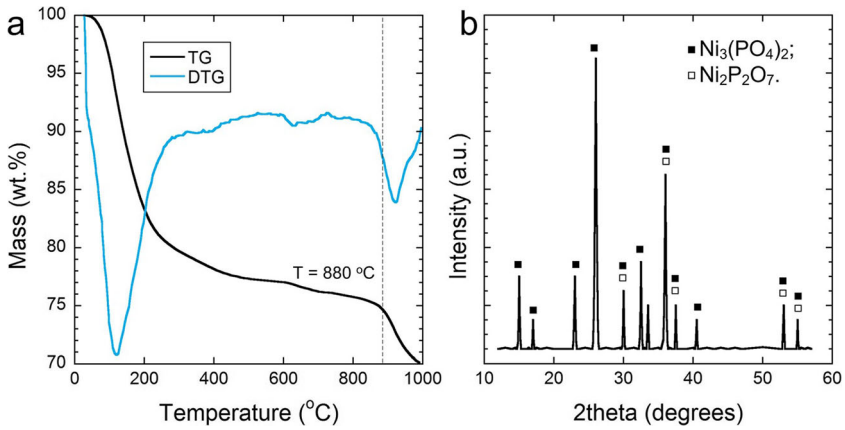

Fig. 4 **a** Thermogravimetric analysis and **b** XRD patterns of the precipitate obtained by the precipitation of nickel ions from the spent nickel-plating electrolyte with sodium phosphate solution after calcination

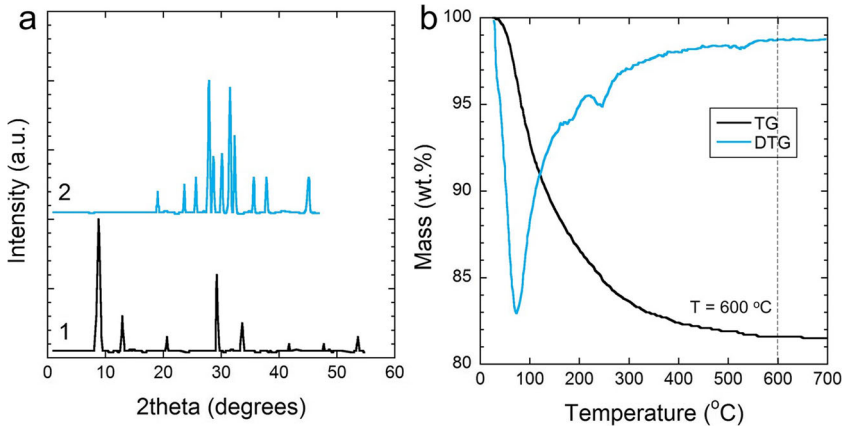

Fig. 5 **a** XRD patterns: 1—after drying; 2—after heat treatment and **b** the thermogravimetric analysis of the precipitate obtained by the precipitation of copper ions from the spent copper-plating electrolyte with sodium phosphate solution

400 °C. At this temperature, black nickel oxide (card #00-044-1159) was formed, which was confirmed by X-ray phase analysis data (Fig. 3a, pattern 2).

When using sodium phosphate as a precipitant for nickel ions, it was found that an excess of the precipitant leads to the formation of nickel phosphate octahydrate and nickel-sodium phosphates, which, assumably, was associated with the incorporation of sodium ions into the crystal lattice of nickel phosphate. Carrying out the precipitation process with a lack of precipitant resulted in the formation of amorphous nickel phosphate, which was confirmed by IR spectroscopy data (Fig. S.1). Based on thermogravimetric analysis (Fig. 4a), the processing temperature of this material was set as 880 °C, for the formation of anhydrous nickel phosphate $Ni_3(PO_4)_2$ of yellow color with an admixture of nickel diphosphate $Ni_2P_2O_7$ (Fig. 4b), cards #00-038-1473 and #00-049-1082.

Synthesis of Cu-based pigments

When copper ions were precipitated by sodium phosphate from spent copper-plating electrolytes, according to X-ray phase analysis, copper phosphate trihydrate $Cu_3(PO_4)_2 \cdot 3H_2O$ (card #00-022-0548) was formed (Fig. 5a, pattern 1). As evidenced by the data of thermogravimetric analysis (Fig. 5b), dehydration of $Cu_3(PO_4)_2 \cdot 3H_2O$ proceeds quite difficult and in a wide temperature range (up to 600 °C). The X-ray diffraction pattern of a sample calcinated at 600 °C shown in Fig. 5a, pattern 2, is characteristic for of anhydrous copper phosphate (card #01-080-0993).

Synthesis of Cd-based pigments

During the precipitation of cadmium ions from the spent cadmium electrolytes by sodium carbonate, a white precipitate of cadmium carbonate was formed (card #00-042-1342). This was confirmed by the data of X-ray phase analysis (Fig. 6a, pattern 1). Heat treatment of cadmium carbonate at a temperature of 400 °C selected based on thermogravimetric analysis (Fig. 6b), resulted in the formation of β-form of cadmium oxide (Fig. 6a, pattern 2), card #03-065-2908, with saturated red-brown color.

Analysis of obtained pigments

To determine the possibility of using the obtained materials as pigments, some of their properties were determined (Table 1), such as oil absorption of the I type, oil absorption of the II

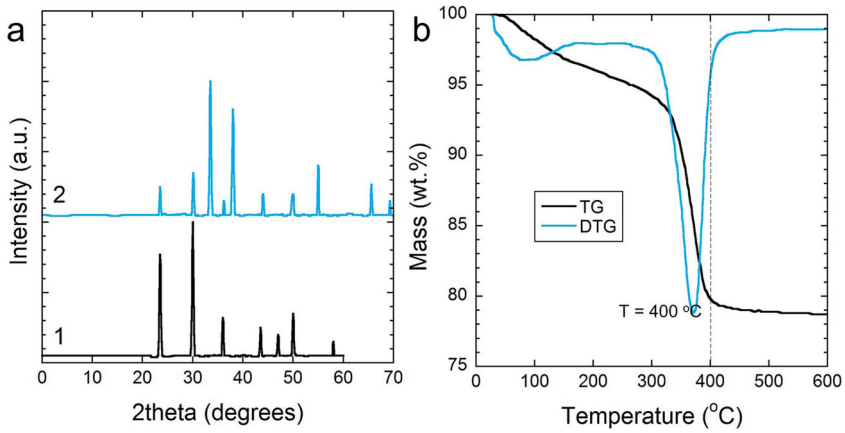

Fig. 6 **a** XRD patterns: 1—after drying; 2—after heat treatment and **b** the thermogravimetric analysis of the precipitate obtained by the precipitation of cadmium ions from the spent cadmium-plating electrolyte with sodium phosphate solution

Table 1 Properties of the resulting pigments

Pigment composition	Oil absorption, g/100 g		Residue after sieving on a sieve no. 0056, %	pH of the aqueous suspension	Color coordinates (L^*, a^*, b^*) for colored pigments, whiteness for whites (%)		
	I type	II type			L^*	a^*	b^*
ZnO	27–33	44–71	0.03	6–7	95–97		
$Zn_3(PO_4)_2$, $Zn_2P_2O_7$	18–34	27–46	0.03	6.5–7.0	95–98		
NiO	19–25	47–53	0.04±0.003	6.5–7.5	22.85–23.42	0.11–0.15	0.88–0.98
$Ni_3(PO_4)_2$, $Ni_2P_2O_7$	46–57	–	0.03±0.004	6–7	64.53–74.15	0.61–1.89	36.54–39.88
$Cu_3(PO_4)_2$	18–22	37–41	0.04±0.006	6–7	64.88–71.08	−8.5–(−13.04)	−63.80–(−68.18)
CdO	15–24	26–39	0.03±0.002	6–7	70.29–75.18	72.25–76.09	60.17–64.11

type, the residue after sieving on a sieve no. 0056, the pH of the aqueous suspension, and the color (Fig. 7).

To define and standardize the color, a three-dimensional model of the CIE 1976 $L^*a^*b^*$ color space was used in this work (Table 1, Fig. 7). For white pigments, whiteness was determined, which ranged from 95 to 98% and meets the requirements for white pigments (ASTM D79 - 86 2020).

The pigments obtained from the investigated spent electrolytes of galvanic production were not inferior in their properties to the pigments currently produced in Russia, China, Germany, France, etc. (Table S.2 and Table S.3).

The resulting nickel pigments have passed pilot testing at a ceramic tile factory. They were introduced into the composition of transparent and muted ceramic glazes for tiles in an amount of up to 20 wt%. Firing was carried out in accordance with the existing technological regulations at a temperature of 1050 °C for transparent glaze and 1200 °C for muffled. In this case, a stable pale yellow coloration of the ceramic glazes was obtained. Zinc oxide was tested as a pigment at the Decorative and Applied Arts Factory. The tests were carried out on a colorless transparent glaze for decorative and applied products (vase). The pigment was introduced in the amount of 10 and 12 wt.%. Firing was carried out at a temperature of 960 °C. A white glaze was obtained. In both cases, grinding of pigments for the preparation of the ceramic glaze was carried out in ball mills.

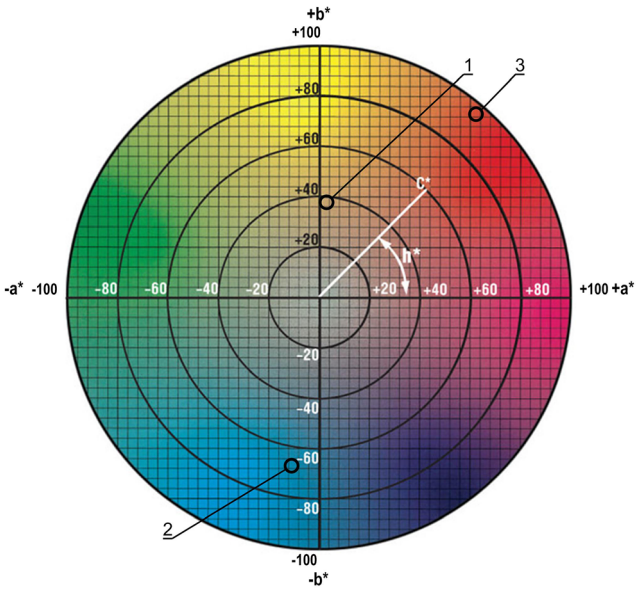

Fig. 7 Areas of coloristic characteristics of samples of composition: 1—$Ni_3(PO_4)_2$, $Ni_2P_2O_7$; 2—$Cu_3(PO_4)_2$; 3—CdO

To the best of the knowledge of the authors, nobody was so far investigated the production of pigments derived from plating electrolytes. Most of the papers discuss different wastes like sludge for the receiving pigments by heating and milling. Plating electrolytes are usually contained only one metal ion comparing with galvanic sludge. Most of them are usually multi-component: ZnMn2O4 (Almeida et al. 2020), Cr/Ni (Carneiro et al. 2019), Al- or Fe-rich sludge (Hajjaji et al. 2010). This fact makes it possible to compare pigments from plating electrolytes with presented ones on the world market.

Technological scheme

Based on the results obtained, a technological scheme for the processing of spent electrolytes was proposed (Fig. 8). The scheme includes the following stages: collection and averaging of the electrolyte composition, dosing of the precipitant into the spent electrolyte, aging of the precipitate under the mother liquor layer separating the precipitate from the solution and washing from water-soluble salts, drying, calcination.

At present, about 110 m^3 of spent zinc-plating electrolytes and 40 m^3 of spent nickel-plating electrolytes

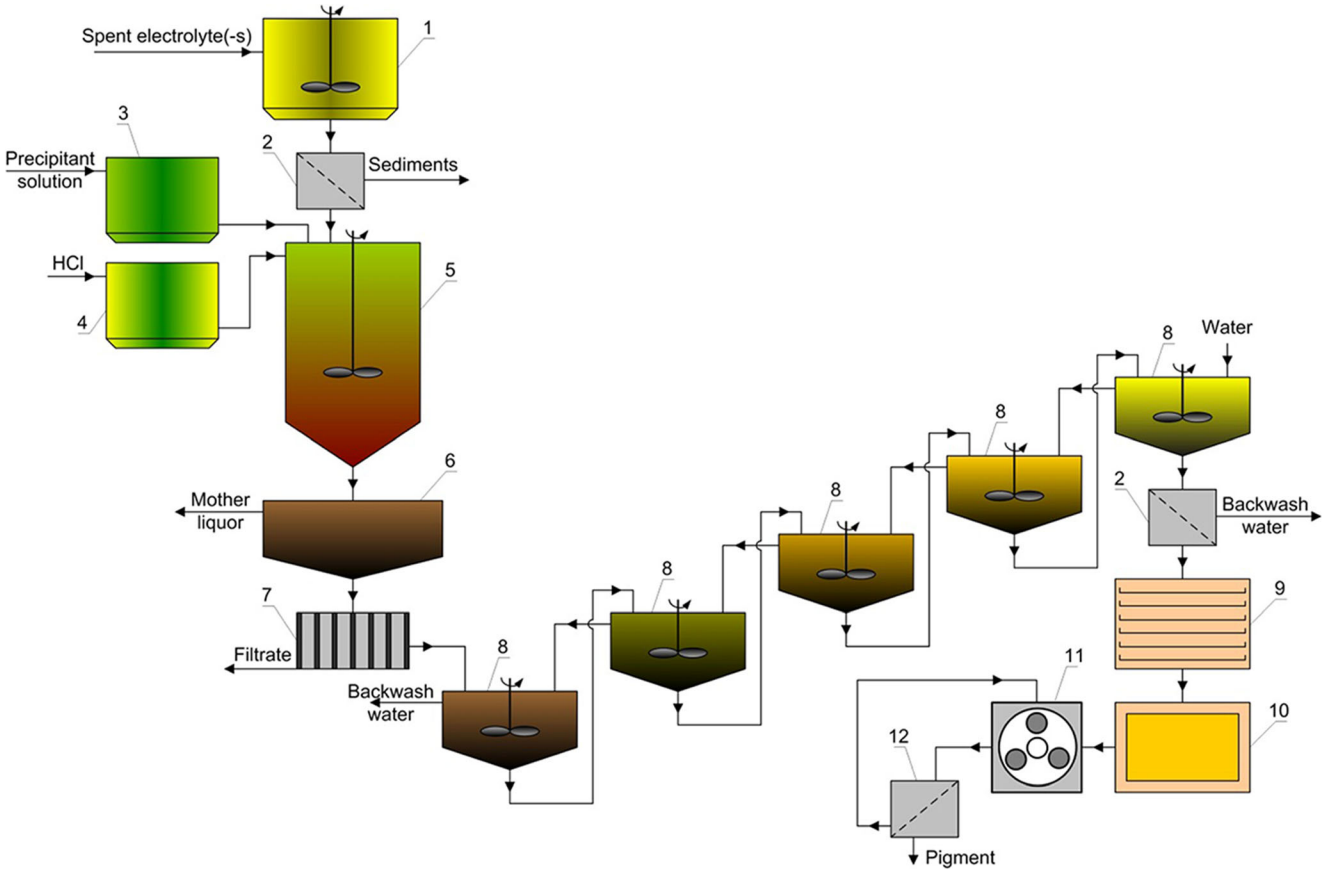

Fig. 8 Technological scheme for obtaining pigments from spent electrolytes of galvanic production, where 1—capacity for averaging of the electrolyte composition; 2—filter; 3—capacity of the precipitant solution; 4—capacity of hydrochloric acid; 5—precipitation reactor; 6—thickener; 7—filter press; 8—rinsing thickener; 9—drying chamber; 10—furnace; 11—ball mill; 12—sieve

are generated in the Republic of Belarus. Their processing according to the proposed technology will make it possible to reduce the import of pigments by 10.8–16.5 tons per year and reduce the amount of generated wastewater sludge from electroplating industry by 10.4 tons (dry matter). At the same time, the economic effect will amount to 27.2 thousand USD per year from the processing of spent zinc-plating electrolytes, and 24 or 5.4 thousand USD per year (depending on the type of precipitant) from processing spent nickel-plating electrolytes.

Conclusion

The paper demonstrates the possibility to use spent electrolytes from electroplating industry as a raw material for obtaining pigments of various colors. The research results make it possible to reduce the load on treatment facilities of the electroplating industry and prevent the release of heavy metal compounds into the environment. The following conclusions could be made:

1. For the deposition of heavy metal ions from spent electrolytes of galvanic production, allowing to obtain pigments next reagents were chosen: for the deposition of zinc ions—sodium carbonate or phosphate, for nickel ions—sodium hydroxide or phosphate, for copper ions—sodium phosphate, and for cadmium ions—sodium carbonate.
2. The conditions for the deposition of zinc, nickel, copper, and cadmium ions from the spent-plating electrolytes and the temperature regimes for processing of the obtained precipitates were determined.
3. The chemical composition, structure, and properties of the obtained compounds had been determined, which confirm the possibility of their use as pigments of various colors.
4. The processing of spent electrolytes according to the proposed technology will make it possible to reduce the concentration of heavy metal ions to acceptable values (0.13–

0.65 mg/L), as well as to reduce the volume of wastewater sludge formed (by 53–115 kg when processing 1 m^3 of spent electrolyte).

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s11356-021-13007-4.

Data and materials availability The supplementary information provides more detailed information about the pigments composition, IR spectroscopy result, properties of pigments derived from waste electrolytes and pigments supplied by manufacturers to the global market, and the additional text.

Author contribution Volha Zalyhina: conceptualization, methodology, data curation, writing—review and editing. Victoria Cheprasova: formal analysis, investigation, data curation, writing—original draft, writing—original draft, writing—review and editing, visualization. Volha Belyaeva: investigation. Valentin Romanovski: validation, formal analysis, data curation, visualization, writing—original draft, writing—review and editing, visualization

Declarations

Ethics approval and consent to participate Not applicable.

Consent for publication Not applicable.

Competing interests The authors declare no competing interest.

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