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Effect Of Calcination Temperature On The Morphology Of Plating Sludge

N. Tugrul, O. Dere Ozdemir, S. Piskin

***Abstract*— The effect of calcination temperature on the morphology of plating sludge generated from waste water treatment plant was studied. This waste was hazardous waste due to high toxic metal content [1]. It must be recovered, reuse or recycled before the process but its properties should determined in order to decide recovery method [2]. Previous researches were shown that this kind of wastes were used in different industries such as ceramic, glass, pigment, etc., at this point, its behavior against to heat was very important to decide the suitable recovery method [3,4,5].**

In this study, the sludge was dried and calcined for 3 hours at temperatures ranging from 650 to 1200°C. Phase transformation of the calcined powders has been investigated as a function of calcination temperature by room-temperature X-ray diffraction and scanning electron microscopy techniques. With increasing calcining temperature, metal oxides were formed cubic spinel phase ($ZnCr_2O_4$) with calcium iron phosphate.

***Index Terms*— Calcination, Hazardous Waste, Morphology, Plating sludge,**

I. Introduction

Globally, governments and private sectors alike are in favor of sustained economic growth as means of providing goods and services to meet human needs in addition to wealth creation. However, this novel drive also brings into effect unintended consequences owing to rapid expansion of industrialization, urbanization, extensive agriculture and rigorous exploitation of natural resources, chief among them being the generation of enormous quantities of hazardous wastes [6].

Industrial wastes often contain potentially toxic chemicals. Improper treatment, storage, and disposal of hazardous wastes can result in contaminant during possible exposures, and potential adverse health and environmental impacts. In general, any chemicals can cause severe health impairment or even death if taken by humans in sufficiently large amounts. A simple model for illustrating the routes of exposure and pathways of toxic chemicals from hazardous waste disposal sites is shown in Figure 1.

Waste characterization is an essential requirement in the development of an effective industrial waste management plan. Prior to establishing strategies for the control and treatment of waste, it is necessary to know whether the waste contains constituents which are hazardous [7].

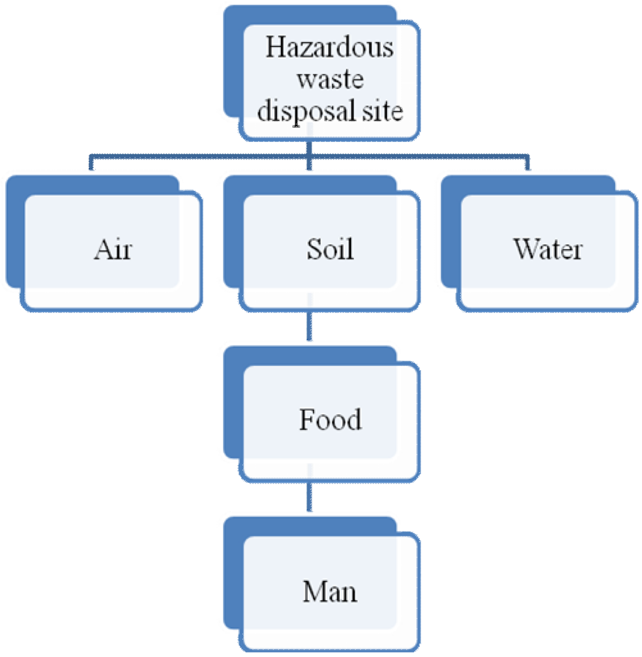

Figure 1. Routes of exposure and pathways of toxic chemicals from hazardous waste disposal sites [8].

II. Materials and Methods

A. Sample Preperation

Plating sludge was obtained from metal plating waste water treatment process. Sample preparation procedure is shown in Figure 2. Firstly it was dried at 105 °C following grounding and sieving.

B. Calcination

Prepared sample was calcinated at 650°C, 850°C, 1000°C, 1200°C at 3 hours (Figure 2)

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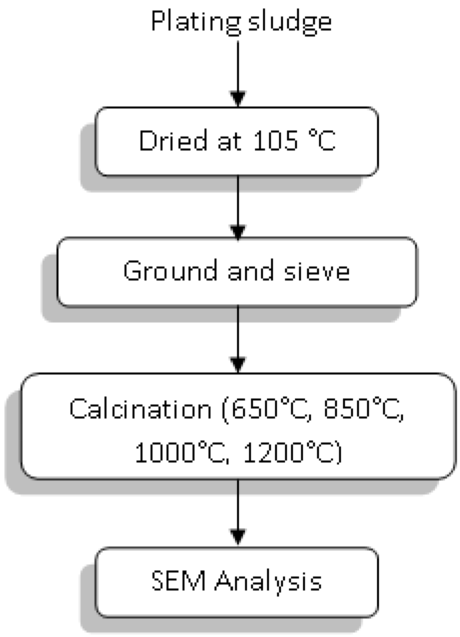


Figure 2. experimental procedure scheme of plating sludge

C. SEM Analysis

The samples calcinated at different temperatures were analyzed with SEM (JEOL, JSM 5410 LV) equipped with backscattered and secondary electron detectors coupled with EDS was employed to observe the morphology of the calcined samples. A gold layer was coated onto the sample's surface for the SEM investigation. The SEM–EDS provides detailed imaging information about the morphology and surface texture of individual particles, as well as elemental composition of samples. The SEM–EDS provides detailed imaging information about the morphology and surface texture of individual particles, as well as elemental composition of samples.

The three most common modes of operation in SEM analysis are backscattered electron imaging (BSE), secondary electron imaging (SEI), and EDS. In this study, BSE and EDS were used to characterize the plating sludge samples. BSE provides visual information based on gray-scale intensity between chemical phases. Backscattered electrons are high-energy electrons that are reflected directly from the specimen surface.

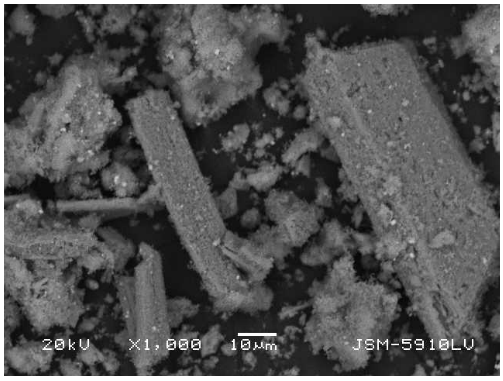
The elemental composition of a sample is determined using characteristic X-ray spectrum of the specimen being examined. The elemental analysis was performed in a ‘‘spot mode’’ in which the beam is localized on a single area manually chosen within the field of view. The EDS detector was capable of detecting elements with atomic number equal to or greater than six. The intensity of the peaks in the EDS is not a quantitative measure of elemental concentration, although relative amounts can be inferred from relative peak heights.

D. X-Ray Difroctometry

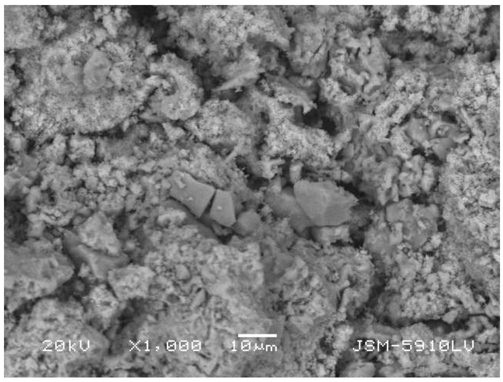
X-ray diffractometry (XRD, Perkin Elmer, X’Pert Pro) using CuKα radiation was used to determine the phase formation.

III. RESULT AND DISCUSSION

The SEM data clearly indicated intermixing of Zn–Cr mineral phases and the predominance of Ca–Fe-P minerals after 850 °C. These results were consistent with original samples’ XRD data, quartz (SiO_2) (ICSD-PDF 085-0795), gypsum ($CaSO4.2H_2O$) (ICDD-PDF 036-0432) and calcium sulfate hemi hydrate ($CaSO_4.0.5H_2O$) (ICSD-PDF 083-0438). In addition calcined samples’ (1200°C) XRD data, zinc chromium oxide ($ZnCr_2O_4$) (ICSD-PDF 073-1962), calcium iron phosphate ($Ca_{19}Fe_2(PO)_{14}$) (ICDD-PDF 049-1223) and copper oxide (CuO) (ICSDPDF 045-0937). One of the part of the particles are consisted of solid spheres at 1200°C (Figure 3 (g)) which is corresponded to calcium iron phosphate entitled number 2 in Figure 3 (d). The other part of the particles is associated with $ZnCr_2O_4$ entitled number 1 in Figure 3 (d). The particles were agglomerated with the increasing temperature.



a)



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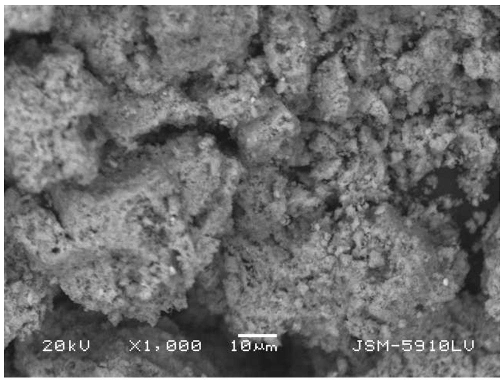

c)

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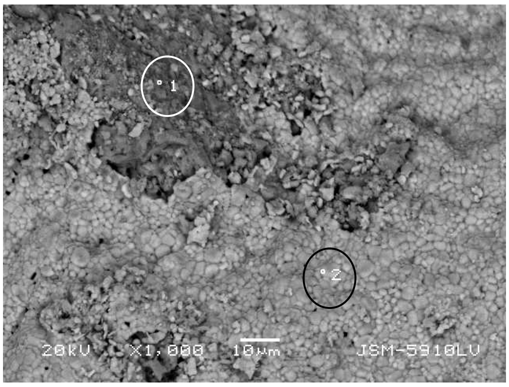
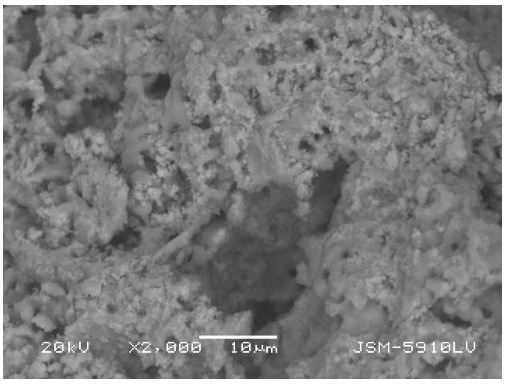


d)



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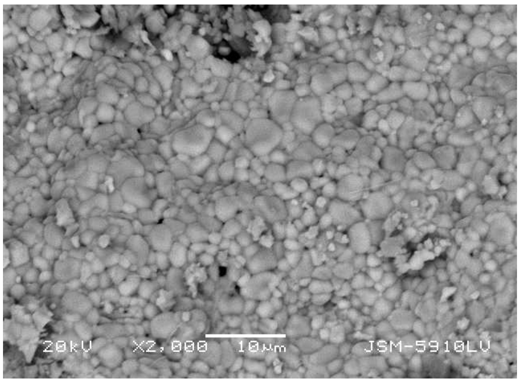

f)

Fig. 3 SEM images of calcined plating sludge, a)650°C, b)850°C, c)1000°C, d)1200°C, e) number 1x2000 magnification, f) number 2x2000 magnification

As determined by EDS, the predominant elements in the plating sludge samples were calcium, iron, phosphate, oxygen, chromium, zinc, and nickel in calcined samples at 1200°C. Iron and phosphate were primarily associated with calcium. In addition chromium and oxygen were associated with zinc. Lesser amounts of the elements, magnesium, sodium, and aluminum were observed with the other elements (Figure 4 and 5).

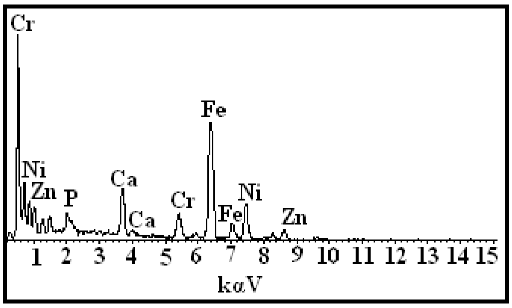

Figure 4. EDS results of number 1 that chosen area

Table 1. EDS results of number 1 that chosen area

Elements	(%)
O	34.25
Mg	2.38
Al	1.79
P	1.15
Ca	4.59
Cr	4.38
Fe	30.84
Ni	13.83
Zn	6.79

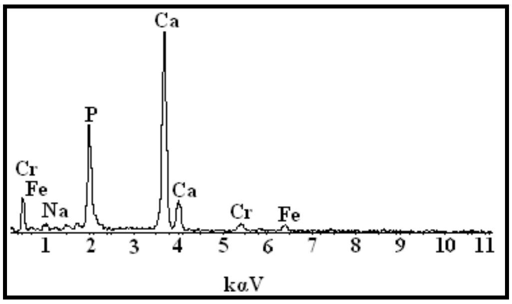

Figure 5. EDS results of number 2 that chosen area

f)

Table 2. EDS results of number 2 that chosen area

Spectrum	%
O	39.15
Na	1.56
P	13.87
Ca	39.35
Cr	2.65
Fe	3.42

IV. DISCUSSION

The chemical and physical properties of calcined samples particles are a function of the mineral matter in the plating sludge and the calcination conditions. During the calcination process, the heat causes the inorganic mineral to become volatile or to react with oxygen. Besides, particles were agglomerated with increasing temperature.

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