

WASTE RECYCLING THE ALUMINUM ANODIZING PROCESS FOR INTERNAL COATING OF OIL REFINING PIPES

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Abstract. *The significant volume of industrial sludge of aluminum anodizing industries has caused great environmental impact. This sludge has a significant amount of aluminum hydroxide that has been heat treat at temperatures of 200°C, 1000°C and 1400°C gives, after X-ray diffraction tests and X-ray fluorescence, gradual increase of the percentage of alumina 93% the end of the last calcinations temperature. With obtaining ceramic body high percentage of alumina, the aim is the production of refractories for oil refining pipe coating, through mass blasting on metal tubing, checking up through scanning electron microscopy and hardness test Vickers the efficiency of this ceramic body as increased alternative life of the oil refining and pipeline alternative to an environmental impasse.*

Keywords: recycling, alumina, petroleum refining pipe

1. INTRODUCTION

The environmental issue is a concern that has mobilized society in the search for alternative solutions such as the reuse and recycling of the growing volume of waste that has been generated mainly by industrial demand for metal source products. Waste are part of the cycles of nature and the economy, but economic progress has made the development is accelerated and the effect of increasing the amount of waste, some difficult to be recycled naturally. The disposal of waste in the air, water or soil, usually produces harmful effects to the environment and to man himself. This problem becomes more visible when it comes to solid waste, because their degree of dispersion is much lower than of liquids or gases, MOMBACH. Waste recycling is an opportunity to transform a major source of spending on billing source or reducing deposition costs. The incorporation of waste in production processes reduces costs and opens up new business opportunities, and reduce the volume of raw material extraction, preserving limited natural resources, KNISS. The industrial waste of aluminum anodizing process when heat treated, is the generation of alumina ceramic with high-purity 93% approximately. The sludge recycling process after the calcination treatment, shows a high percentage of alumina with high purity of 93% strength approximately the feedstock being used in various segments of the ceramic industry. However, the purity of the alumina is that it will qualify for their use in engineering at least 80% (mostly at least 90%) and Al₂O₃ porosity limit. In practice, the impermeability under ambient conditions requires that the total porosity is less than about 6%, RVSHKEWITCH and RICHERSON. The alumina ceramics are most commonly used where there is the need for a high mechanical resistance, being classified as special ceramics. The paper aims, the inner lining of the oil refining pipe in order to ensure longer life time and consequently effective security in the oil refining, avoiding the risk of accidents. Crude oil is a very corrosive and chemically reactive product due to the presence of constituents such as SO₂, leading thus to surface degradation of the oil refining pipes are made of metallic materials, cause accidents. Thus, an option to resolve this problem is to use an inert coating that corrosion and chemical attack.

2. METHODOLOGY

The development work has met the solid residue collected in the steps of anodizing and heat treating industry, such as oven drying product generating MF 01 - 200 °C and calcination in high temperature furnaces generating product 02 MF - 1000 °C; MF 03 - 1400 °C.

2.1 X-Ray Diffraction

For the execution of the X-ray test, we used a Shimadzu XRD-6000 diffractometer with Cu radiation, $K\alpha = 1.54060\text{\AA}$, a scan $10^\circ < 2\theta < 100^\circ$. The X-ray diffraction provides a convenient and practical means for the qualitative identification of the crystalline compounds DA SKOOG.

2.2 Scannig Electron Microscopy (SEM)

Microanalysis is one of the most important instruments for chemical analysis of organic and inorganic materials. Through the identification of X-rays is possible to determine the chemical composition of regions with up to $0,1\mu\text{m}$ in diameter. It is a non-destructive technique capable of producing high magnification images (up to 300.000x). Because the image presented by the SEM display three-dimensional view, you can get answers as pore size and evaluate the surface structure of the sample grain studied.

2.3 Vickers Hardness

For the analysis of microhardness of the samples before immersion in the oil were used Shimadzu microhardness tester, Model HMV-2, with application of a load of 2942 N for 30 seconds in four random points of the sample surface by a Vickers indenter. After immersion in crude oil use a microdurometer coupled to a Zeiss microscope metallographic, Jenavert model penetrator HVS-5 Model No. 0021 with application of a 1000gf load for 10 seconds, made 7 indentations and eliminate the two most dispersed, getting five results hardness for each sample that had contact with the oil of land and sea for 30 days.

2.4 Optical Microscopy (OM)

Through this analysis you can check details of the surface of the sample being studied as potential defects, imperfections. In this work we used an optical microscope Olympus BX 51, where it was possible to analyze the surface before and after immersion in crude oil and thus evaluate the results. Samples MF 03 - 1400 °C were immersed in oil for 30 days with the purpose of the analysis of the same surface when exposed to conditions of a highly corrosive product.

3. RESULTS AND DISCUSSION

The drying in an oven at a temperature of 200 °C for 24 hours sludge which has grayish white color, and after leaving the oven acquired light brown color according to Fig. 1 (A and B). The 11 sludge samples drying recorded a significant reduction in mass with loss of moisture of 88%, as shown to Fig. 2. The calcination treatments were performed at temperatures of 1000 °C and 1400 °C for periods of 24 hours and 48 hours, respectively.



Figure 1 - (A) sludge before drying in an oven; (B) dry sludge kiln.

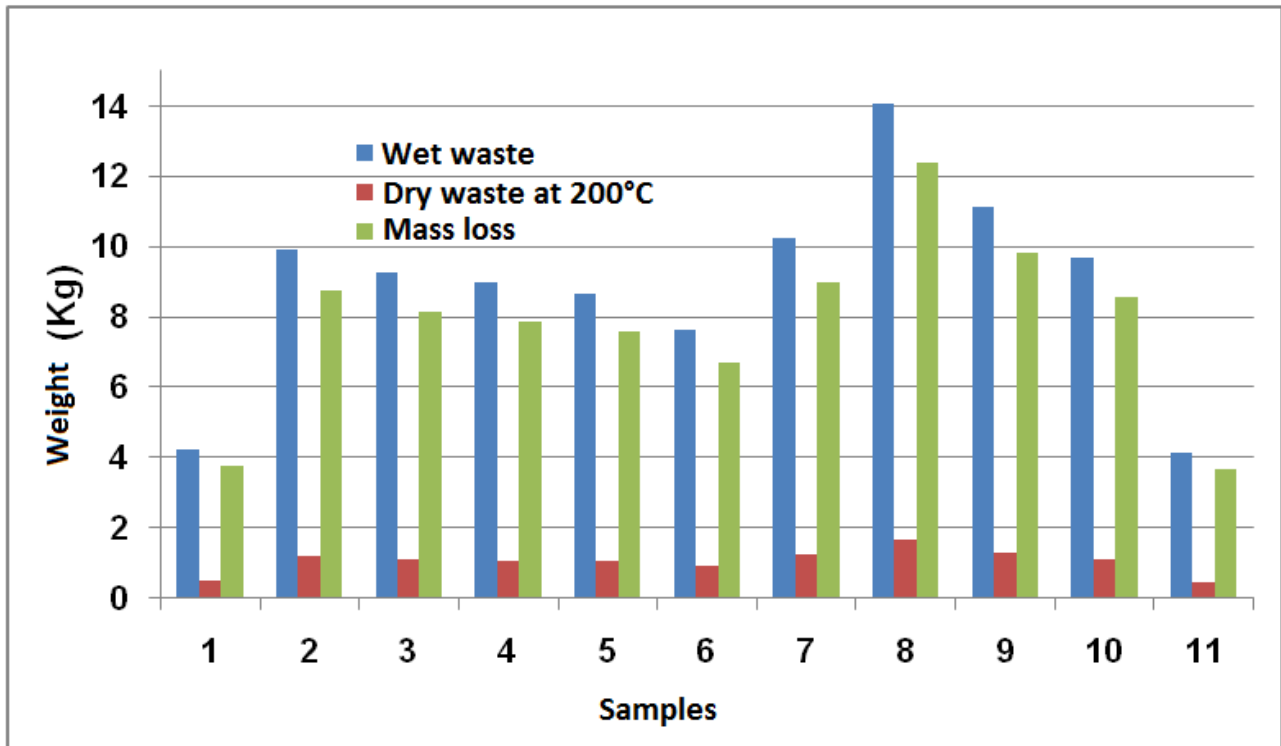


Figure. 2 - Distribution Graph drying of the waste at 200 ° C.

Later obtained the characterization of physical and chemical properties, always the guidelines of national and international regulatory standards for the characterization of materials. The materials related to the sample 03 MF was ground in MA-500 Ball mill for twenty-four hours, generating a fine particle size. After milling waste benefited in sieve # 200 mesh, MOMBACH. Specimens were made with 100% residue (calcined at 1400 °C (MF03)). The molding was uniaxial load of 12 tons on metal mold 30mm in diameter and 05mm thick. These specimens after sintering resulted in well-resistant tablets and underwent the intended technological tests in research, as shown in Fig. 3.



Fig. 3 - The sample test body 03 MF sintered at 1.400 °C.

3.1 X-Ray Diffraction

The XRD spectrum of the sample is MF03 shown in Fig. 4. In general the most intense peaks recorded in X-ray diffraction chart, are preserved registered as corundum. Enroll exogenous phenomena crystal compound destructuring volatile chemical element firing to 1000 °C and this fact is intensified with the increase of over 400 °C in the calcining thereof, as illustrated in the Fig. 4 and 5.

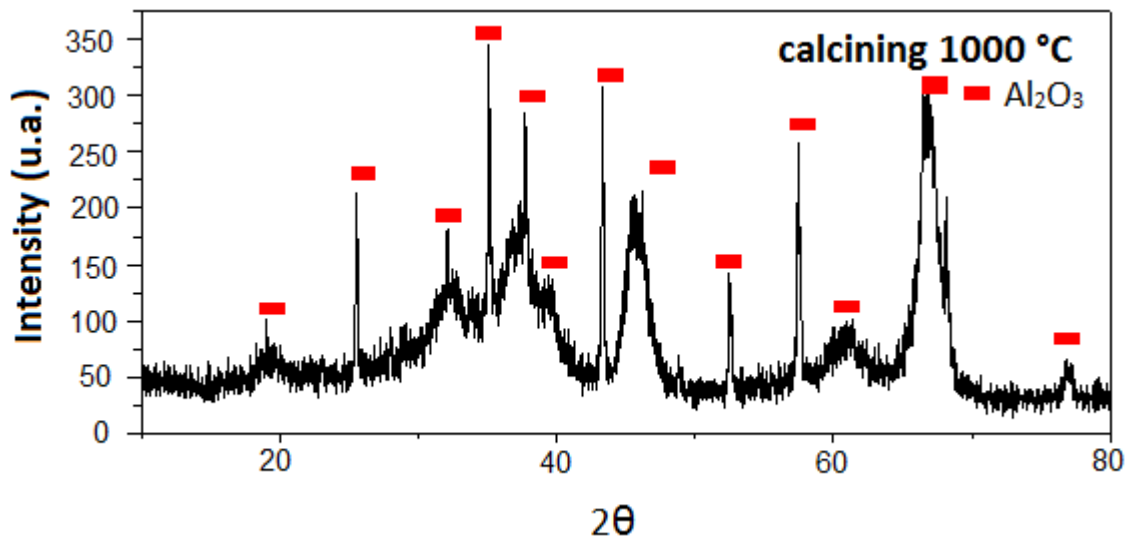


Figure 4 - XRD of calcined residue at 1000°C.

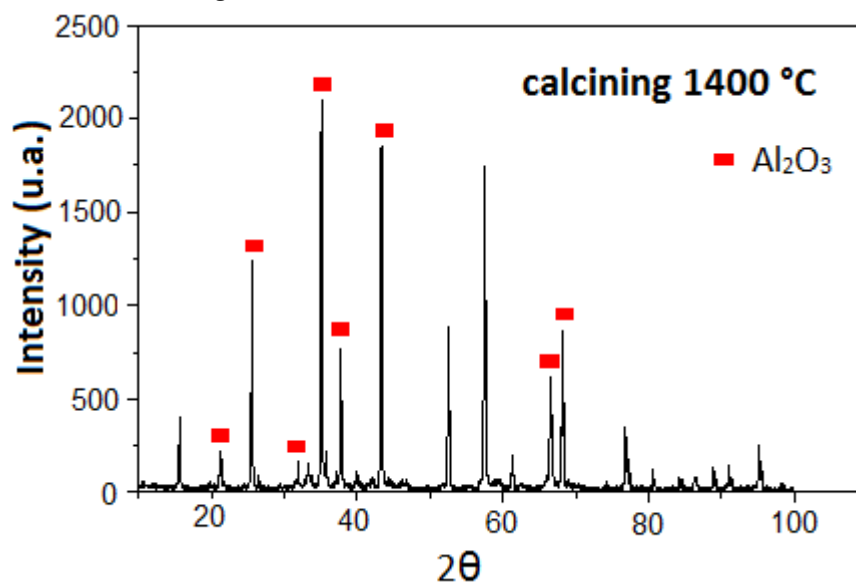


Figure 5 - XRD waste heat treated at temperature of 1400°C.

3.2 Electron microscopy analysis (SEM)

The micrographic images showing surface morphology with predominantly particle size and well defined grain boundary as a result of the effect of heat treatment. In Fig. 6 (A) of the enhanced image 15KVx1000 (10µm) ceramic mass after 48 hours burning at a temperature of 1.400 °C is presented with well distributed pores and in many sizes. A predominance of size of particles in the form of plates which present with good adhesion between them, which can influence the mechanical strength property when submitted to physical efforts. In Fig. 6 (B) in detail, increased, there is the formation of grain boundaries in the particles and the porosity of the material with the bonding between the particles occurred during the calcination process 1400 °C.

In Fig. 7 (B), arrows point the ligament particles and formation of grain boundaries between them was at sintering process at 1400 °C. It appears more clearly that the particles of the specimen surface of the ceramic mass MF03 register, so a formation on plates with angular and rounded particles of various sizes of crystals sintered contours.

In sintering, the trend is that the entire inner surface of the workpiece disappears and it becomes quite dense. It actually only occurs if the temperature is high and the time is long enough to occur transformations of matter.

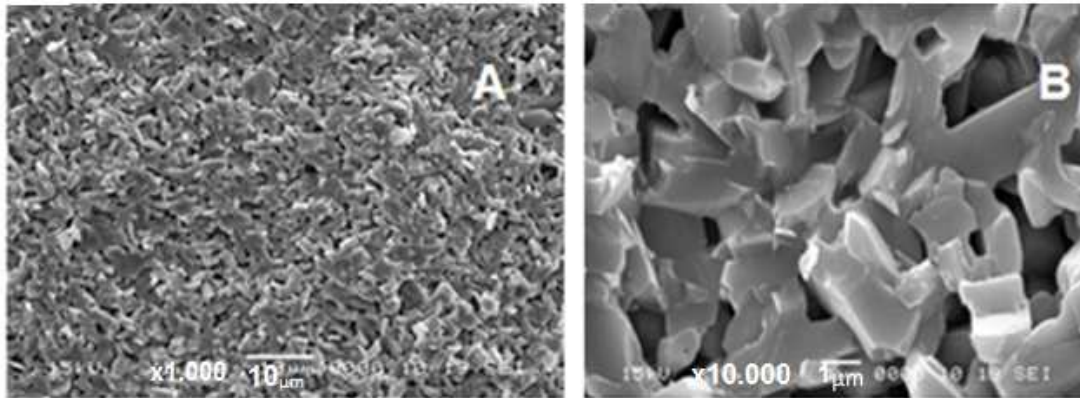


Figure 6 – (A) and (B) Electron microscopy analysis

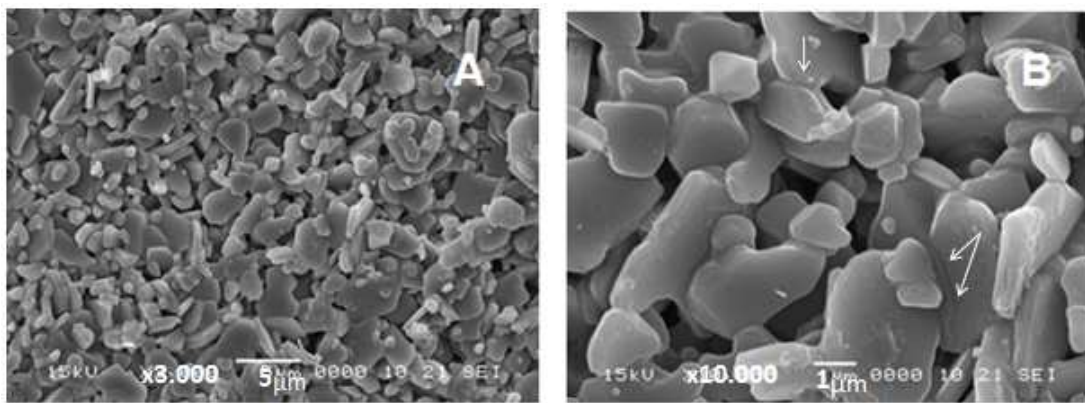


Figure 7 - (A) and (B) Electron microscopy analysis

3.3 Analysis of EDS

The justification of importance to the EDS analysis is checking the homogenization of the chemical elements that make up a ceramic body. It sought to verify the distribution of chemical elements within the specimen sintered at 1400°C. Since the resistance of a ceramic body is completely the question of intrinsic chemical elements that may favor the growth of grain boundary and consequent reduction of porosity during firing. The ceramic specimen of mass MF03 (Fig. 8-A) had a fracture submitted to the analysis of EDS (Fig. 8-B).

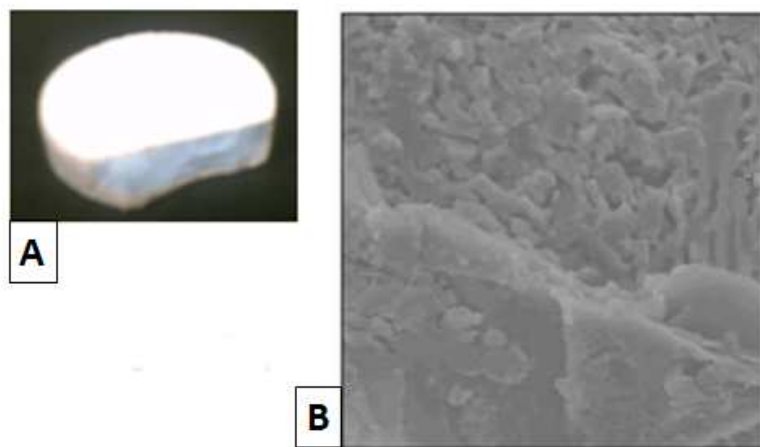


Figure 8 – (A) ceramic specimen (B) analysis of EDS

They were recorded intense peaks of Au (gold) and Al (aluminum) in the EDS test. Gold is coming from the metallization process of the sample for analysis and Al (aluminum) is the most abundant element in the chemical

composition of the sludge as raw material. The element Si (silicon) contained within the chemical composition of the ceramic body because it has intrinsic relations with the bauxite ore.

3.4 Microhardness analysis

In order to relate the physical characteristics with the mechanical properties of the specimens in the calcined mass MF03, the hardness analysis with four point measurements in ceramic wafer following the criteria of the circle quadrants was held. In this analysis after applying 2942 N load for 30 seconds at each point it was possible to register hardness resistors (HV) with different variations between 283HV, 288HV, 302HV and 308HV. Mean ($\bar{X}$) 295.25 was obtained and the uncertainty of the result of measurement expressed as the standard deviation measured 10.13. The importance of this technological considered test relates to the events of impacts, and physical wear to which the coating on the pipe will be exposed during its lifetime.

3.5 Optical Microscopy (OM)

The samples were immersed in oil for 30 days analyzing the images obtained by light microscopy, Fig.9, there is no significant change on the surface of our samples in direct contact with crude oil, ie, cracks were observed or any evidence of chemical attack by the oil during the 30 days immersion, which indicates a good chemical stability for the period adopted and raise the status of an inert material.

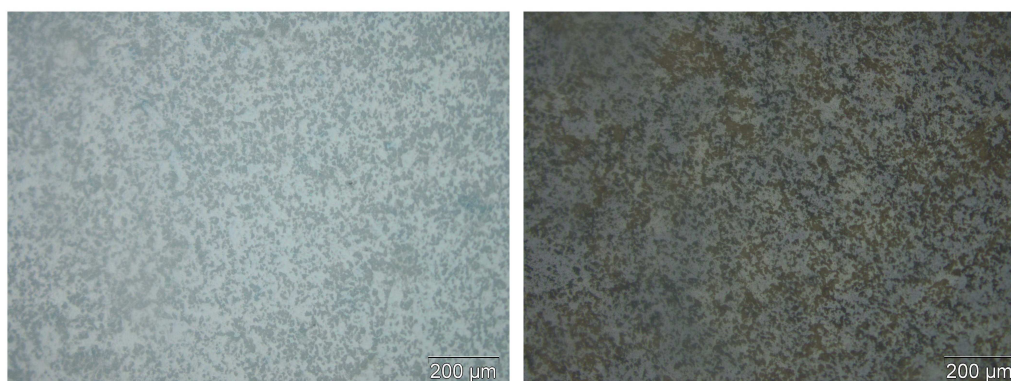


Figure 9 - MF Optical microscopy of the sample 03 before and after immersion in oil for 30 days.

4. FINAL CONSIDERATIONS

In the analyzes conducted to characterize the chemical and physical properties of industrial sludge anodization of metallic profiles ALCOA-PE was found that their reuse is possible without the use of additives.

The particle size of dry sludge state is much thinner than the particle size of alumina originating from an industrial process for the beneficiation of bauxite ore. The fineness of the ceramic body provides lower porosity, higher mechanical strength of the ceramic end product.

The ceramic mass developed called MF03 reported a very low water absorption level. Therefore they can not be considered restrictions on the use in pipe coating oil refining MF03 ceramic body. The calcination temperature of 1400°C is beneficial for effective bonding between the particles during 48 hours. The average particle uniformity rating ensures that have less space between the particles and consequently results in fewer pores within the ceramic body after calcination. Sintering at 1400 ° C gives the concentration of metals and crystalline destructuring reorganization of some minerals contributing to a better organization of the ceramic body and strength gain.

This work shows, through the results, good potential for recycling the residue analyzed, obtaining after the tests performed and observed the production of alumina with 93% purity in the semi-quantitative chemical analysis. The results of the experiment indicate that the line of research on a slurry anodizing industry starts with the heat treatment, and proposed for coating production line for the oil pipes.

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