Journal of Chemical Technology and Metallurgy, 60, 5, 2025, 799-808

STUDY ON RECOVERY OF ALUMINA GRAINS AND FIBERGLASS FROM REJECTED AND USED ABRASIVE DISCS

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Received 28 January 2025
Accepted 21 March 2025
DOI: 10.59957/jctm.v60.i5.2025.11

ABSTRACT

A method was developed for the quantitative regeneration of Al_2O_3 from rejected and spent abrasive discs by pyrolysis. It overcomes the shortcomings of the method described in the literature, namely, the pyrolysis of the abrasive waste takes place without its preliminary crushing and, the pyrolysis takes place at the same speed on the surface of the disc as in volume. This method allows recovering of alumina, fiberglass mesh and cryolite. SEM images of regenerated and virgin Al_2O_3 show similar sharp edges. The results from the elemental analysis revealed that the content of elements in virgin Al_2O_3 and in the recovered one are the same. Size and orientation characterization of recovered Al_2O_3 and virgin Al_2O_3 is reported. The recovered alumina was used for the preparation of cutting abrasive discs. The examination results revealed that all tested disks meet the corresponding standards

Keywords: abrasive discs, waste recovery, aluminium oxide, phenol formaldehyde resin, pyrolysis.

INTRODUCTION

Recycling of waste materials is one of the most significant global problems of the World, because it is connected to the storage of raw materials and energy, and environmental protection. The waste in the production and especially the use of abrasive discs deserves special attention, as the amount of the used abrasive discs is huge due to their widespread application in automotive and transportation, heavy machinery, metal fabrication, construction. In 2020, the worldwide abrasive market was estimated to be worth USD 34.41 billion and is expected to grow at a compound annual growth rate of 4.2 % from 2021 to 2028 [1]. Fabrication waste from abrasive disks alone amounts to 25000 tons annually. Abrasive fillers make the largest contribution to the value of the disks as their value varies from 500 euro per ton for brown corundum's to 40000 euro per ton for sol-gel grains. Therefore, the fabrication waste recycling market alone can be estimated at about 12.5 million euros per year [2].

On the other hand, fiberglass is even more valuable material. The price of fiberglass mesh varies from 1000 to 1500 USD for 100000 pieces. From this point of view the recycling the abrasives improves the process' efficiency, economy, and environmental friendliness. Disposal of such solid waste materials in the environment results in serious environmental problems such as land, air and water pollution. Improper disposal of solid waste leads to soil pollution and results in health risks, soil contamination and ecological risks [3]. The rejected or spent wheels are disposed of as solid waste. Each year, over 52181640 pounds of spent grinding wheels are dumped in landfills [4, 5]. This is a loss of valuable mineral. Based on the literature review, the methods for recycling of rejected and spent abrasive discs can be divided into the following processes: mechanical; thermal; chemical and biological related recycling. The recovered abrasive material because of mechanical crushing changes the particle size which is decisive for its efficiency. The recovered abrasive material contains also broken fiberglass mesh and the organic binder - bakelite [6]. Grains are recovered using mechanical crushing and sieving processes. The recovered grains are used for production of resinoid cylindrical grinding wheel of 350 mm outer diameter [5]. Grains generated from spent vitrified wheels can be beneficially used in resinoid grinding wheel application. Abrasive industry waste was used as a replacement material in the fireclay brick production which can be used in the iron, steel, petrochemical, cement, and glass industries [7]. The replacement of alumina abrasive waste in the fireclay brick results in value-added material and it leads to reduction of the fireclay cost. Recycled alumina abrasive grains were recovered by applying the mechanical crushing method and are reused as abrasive grain in the abrasive water jet machining process [4]. According to the authors, the marble and granite cutting industries' rough cutting operations are most suited for recovered alumina. The separation process follows various methods like, physical [8], chemical [9], magnetic and gravimetric processes [10]. The chemical method is the mostly used in the separation process due to its efficiency rate and the purity of the recovered material.

Chemical separation method was employed to remove the bond from the grinding wheel. To overcome the usual problem of agglomerates in recovered grits, a modified ball milling method is proposed. Brown alumina abrasives can be recovered from vitrified alumina grinding wheel trash by crushing the grains and utilizing hydrofluoric acid as a chemical agent, according to a method proposed by Sabarinathan et al. [11]. The crushing process leads to agglomerates, and the leaching process leads to bond removal in the form of sludge. The parameters are optimized for effective grain recovery. The recovered grains' characteristics, like their friability, are contrasted with those of a standardized, controlled grain sample. The presence of elements in the sludge and the presence of agglomerates in the crushed wheel is observed using scanning electron microscopy and energy dispersive X-ray analysis. Singh et al. have shown that grinding waste can also be used as a raw material for the powder metallurgy process [12].

A chemical process for the recovery of all or some of the abrasive elements contained in an abrasive material, wherein the abrasive elements are dispersed in a resin with at least one phenolic hydroxyl group was developed by Goettmann et al. [2].

A process for recovering abrasive grains from vitreous bonded materials by boiling the material in aqueous solution of an alkali metal base is described by Trischuk et al. [13]. Gusse et al. were the first to show the biodegradation of phenol-formaldehyde polymers, providing a basis for exploring bioremediation and biorecycling of phenolic resins [14]. Three separate lines of evidence confirmed their ability to biodegrade using the white-rot fungus Phanerochaete chrysosporium. A colour change in the growth medium (from yellow to pink) suggested that initial biodegradation of the resin occurred three days post-inoculation. The removal of phenol-formaldehyde resin and embedded abrasive grains from coated abrasive disc is used a sandblasting technique [15].

In the case of a thermal process, the recovery of the abrasive material is a result of the thermal decomposition of the organic binder. Pyrolysis provides an opportunity for the recycling of abrasives and is therefore attracting increasing attention. Liquid, gas, and char are the 3 major products of pyrolysis. In addition, the pyrolysis is not expensive to implement and is possibly located near the feedstock source. Giani et al. have developed a method for recovery of abrasive grains by pyrolysis [16]. The method involves the following stages: 1) gathering waste that contains residues; 2) mechanically treating the residues with abrasive grains obtained in (1); 3) thermally breaking down the mechanically treated abrasive grain residues from (2); 4) breaking up the thermally processed abrasive grain residues from (3); 5) isolating the broken-up abrasive grains from (4) and separating them from other materials. The process of thermal material breakdown includes both pyrolysis and oxidation, which occur in step (3) at temperatures commencing at 400°C. The optimal temperatures for achieving the best outcomes fall within the ranges of 450°C to 750°C and specifically from 500°C to 600°C. Goettmann et al. comment the thermal process and conclude that: i) the thermal approach requires high temperatures (600°C) for degradation of organic binder; ii) the abrasive grains are polluted by the presence of coke, which prevents their reuse. These conclusions are not supported by any data [2].

Data from the literature related to the recovery of waste from the production and application of abrasive discs give reason to confidently claim that pyrolysis has significant advantages over the other described methods, namely: (i) the abrasive material, the glass mesh and cryolite; (ii) as a method, it is much safer compared to chemical regeneration methods, which use aggressive acids and bases, posing a danger to human health and the environment; (iii) readily feasible in industry; (iv) can be a waste-free technology.

The aim of this article is to investigate the recycling capabilities of the thermal process (pyrolysis) for abrasive discs with organic binder, aiming at the regeneration of the abrasive grain, cryolite and the fiberglass mesh, and possibilities for their reuse.

EXPERIMENTAL

All abrasive dicks used in this study were provided by the Abrasive Technology Ltd factory, Berkovitsa, Bulgaria. Abrasive disc with weight 64.5 g - A 24- R-BF metal-metal, diameter 125 mm consists of Al_2O_3 - 48.0 g, (74.4 %); Cryolite - 5 g (7.7 %); liquid resin (resol) - 3g, (4.6 %); solid resin (novolak) - 7 g, (10.8%), fiberglass mesh 1.5 g (2.3 %) were used.

Their processing begins with heating to 650°C for about 30 min (pyrolysis time) in a muffle furnace Apro Term M 9 L. After cooling the furnace and the regenerated abrasive material for 30 to 60 min, it was found that the disk was completely degraded. The residue after the decomposition was abrasive materials - alumina, fiberglass mesh and grey cryolite powder. The fiberglass mesh was completely preserved, and it was removed mechanically. The mixture consisting of Al_2O_3 and cryolite was washed several times with water to remove the cryolite. Pure Al_2O_3 was dried at 100°C, while cryolite was precipitated from the wash water and then was dried, too.

Thermogravimetric and chromatographic analysis of abrasive discs before pyrolysis was made by Perkin Elmer TGA4000 hyphenated thermogravimetric analysis and gas chromatography mass spectrometry (TG-GC/MS) system and working conditions as follows: argon flow - 60 ml min^{-1}; temperature program: from 50^{o}C to 920^{o}C - step 15°C min; at 920^{o}C - holding for 3 min.

The recovered alumina (R-Al_2O_3) oxide was characterized by Scanning Electron Microscopy (JEOL JSM 6390) and Energy Dispersive Spectroscopy (INCA Oxford), X-ray Diffraction Methods (Diffractometer Empyrean manufactured by Malvern Panalytical) and X-ray computed tomography methods (SkyScan 1272 manufactured by Bruker).

From the regenerated aluminium oxide, new metal cutting discs were made. The composition for making a new disc contains 48 g regenerated $Al_2O_{3,}$ 5 g cryolite, resin XF 1504P - 7 g; resin XF 5004 L - 3 g; fiberglass mesh type 380 / 123x23, 1 pc; fiberglass mesh type 191 / 123×23CN, 1 pc; metal sleeve - 1 pc; label - 1 pc. The weight of the final product is about 65 grams. The production and testing of these discs follow the same procedure as for discs made of standard alumina oxide. Disc size: 125/3 mm.

RESULTS AND DISCUSSION

Thermogravimetric analysis (Fig. 1) shows that the sample of abrasive disc starts to lose weight at 100°C (~ < 1 %-humidity). Weight loses are ~ 4 % within the temperature range of 550 - 600°C. In this range the decomposition rate is higher. At 900°C the weight loss is 7.62 %.

TG-GC/MS analysis of abrasive disc revealed that the pyrolysis of the abrasive disc metal-metal A 24 R-BF releases methane, carbon dioxide (on two stages) and traces of hydrocarbons. Experiments were conducted where the discs were placed horizontally on top of each other in the pyrolysis reactor. It was found that in some of the inner discs pyrolysis had not proceeded quantitatively, and the duration of the process had to be extended. To avoid this, experiments were conducted where the discs were placed vertically in the pyrolysis reactor. It was established that when pyrolysis is carried out at 600°C for 30 min the organic bond is completely decomposed and the grains, cryolite and glass mesh

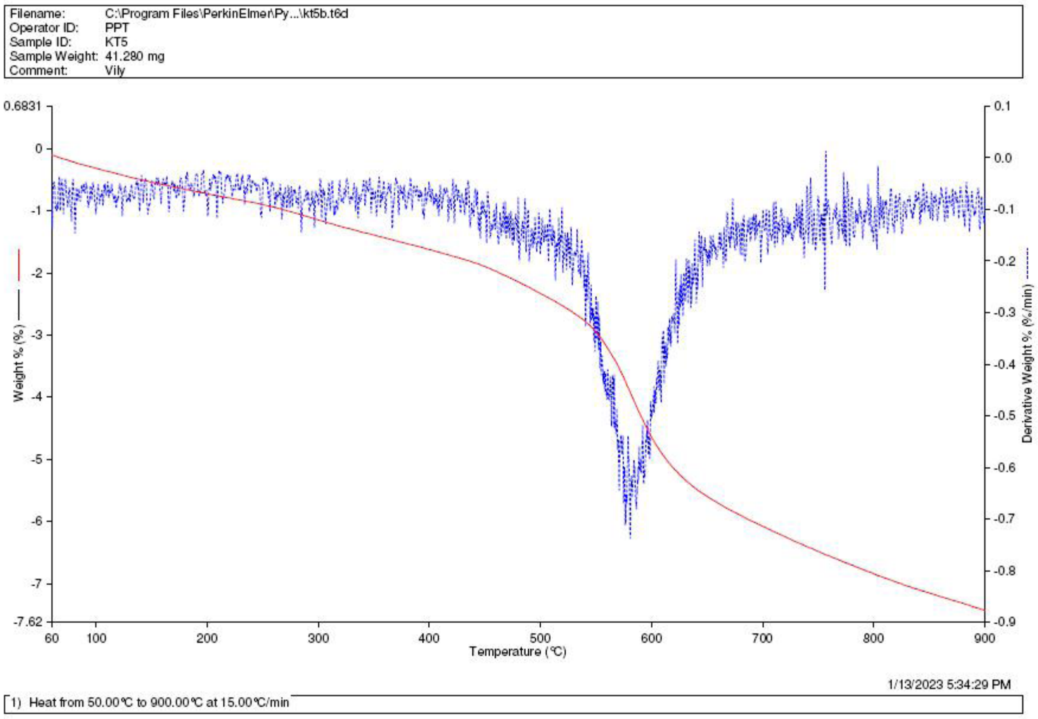

Fig. 1. Thermal gravimetric analysis of the abrasive disk sample.

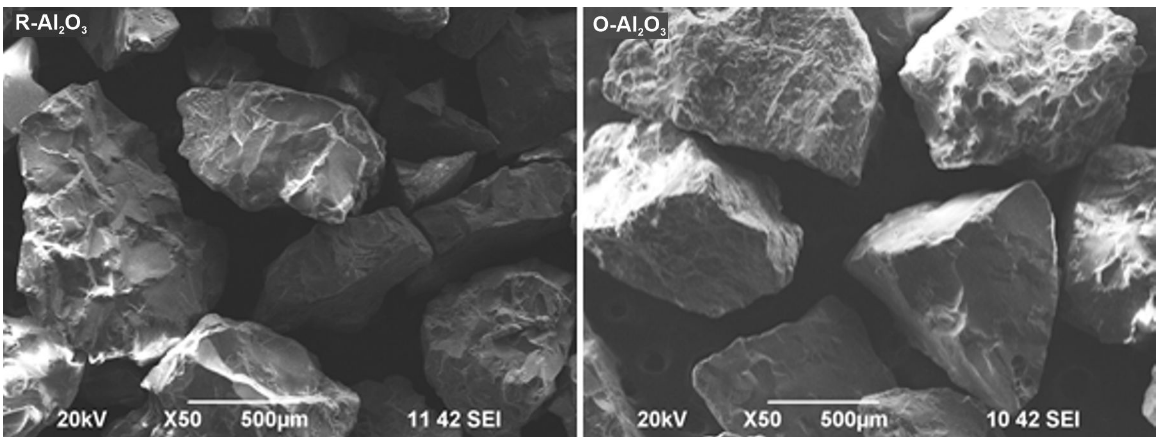

Fig. 2. SEM images of the recovered and virgin Al_2O_3 (magnification x50).

are separated. The recovered Al_2O_3 was washed several times with water and dried at 100°C.

Scanning electron microscope (SEM) is used to study the changes in size and shape of grains after recovering. SEM images of regenerated and virgin Al_2O_3 are shown in Fig. 2.

Similar studies were conducted by Singh et al. [17]. Both samples show similar sharp edges. In the recovered grains, in most cases, the sharp edges of the grain are retained. In general, the recovered grains show no major difference in appearance. The results from the elemental analysis (Table 1) revealed that the content of the elements in the virgin Al_2O_3 and in the recovered one is the same. In recovered alumina oxide, there are 1.29 % of fluorine and 0.16 % sodium. The presence of these two elements is due to the traces of cryolite in the recovered alumina oxide. Angle distribution of recovered Al_2O_3 and virgin Al_2O_3 (Fig. 3) revealed that in both cases the maxima are between 80 and 120 degrees, but for recovered (R-Al_2O_3) alumina the tail is slightly

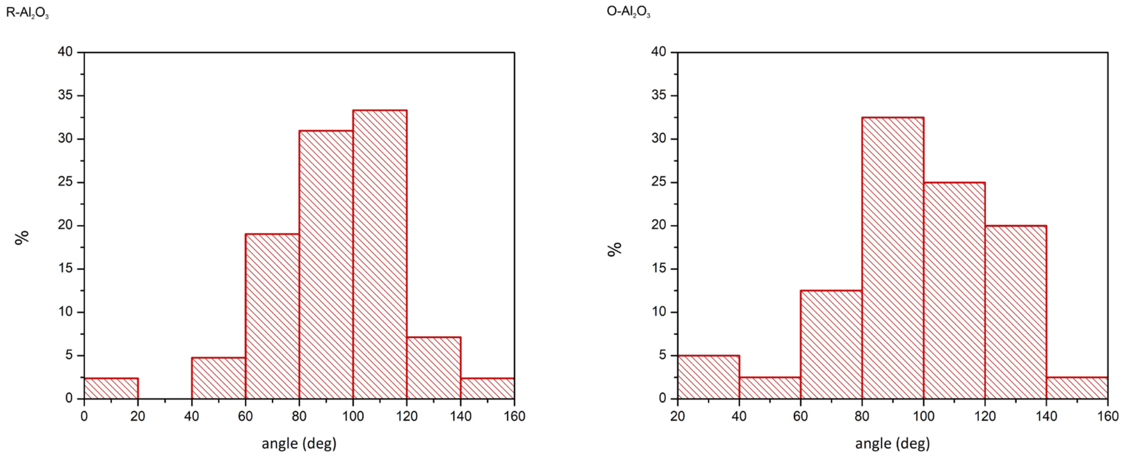

Fig. 3. Angle distribution of regenerated Al_2O_3 and original Al_2O_3.

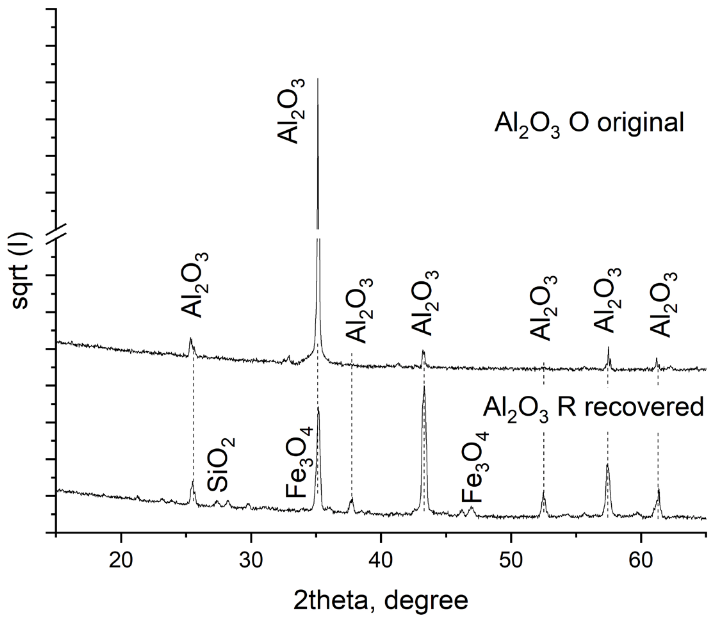

Fig. 4. Diffraction patterns of original O-Al_2O_3 and recovered R-Al_2O_3.

Table 1. Elemental analysis of virgin Al_2O_3 and recovered Al_2O_3 In weight %

	C	O	F	Na	Al	Si	Ca	Ti	Fe
Original Al_2O_3	2.41	49.84	0	0	46.86	0.68	1.02	1.14	0.55
Recovered Al_2O_3	2.46	49.50	1.29	0.16	45.71	0.75	1.20	1.14	0.52

larger than in the original (O-Al_2O_3) alumina, i.e. there is a slightly more rounded particles or at least part of them.

The peak of the distribution of the particle size for recovered R-Al_2O_3 material is between 500 - 800 μm. The distribution is narrower, i.e. the particles are more uniform in size, while for the virgin O-Al_2O_3 the dimensions are generally between 500 and 1000 μm, almost uniformly distributed. The results for the angle and size distribution of recovered and virgin alumina are quite similar.

The phase composition of the original alumina (O-Al_2O_3 and recycled (R-Al_2O_3) is represented in Fig.4.

X-ray diffraction method revealed that in virgin material the main component is alumina, while recovered alumina contains traces of magnetite (Fe_3O_4), and of quartz (SiO_2).

The transformation of hematite into magnetite can be explained by the fact that the samples are heated in a reduction atmosphere in the process of recycling them. The reason for this is that the initial process of reduction of hematite in magnetite starts a little over 400°C. The diffractograms of R-Al_2O_3 show the presence of additional phases of not very clear origin. The probable reasons for their appearance can be the (i) reaction of iron oxides with quartz which is also quite chemically resistant; (ii) reaction of iron oxides with residues of binders. A significant change in the intensity distribution of the corundum phase is observed due to preferred orientation by plane with Miller index (104). After heating this effect diminishes.

The differences of two samples labelled O-Al_2O_3-virgin, and R-Al_2O_3-recovered were analysed with X-ray microtomography. The powders were mixed with soft paraffin in the form of balls and scanned together under the same conditions.

Fig. 5 shows typical specimen cross sections. The particles of sample R are smaller, and the particles of

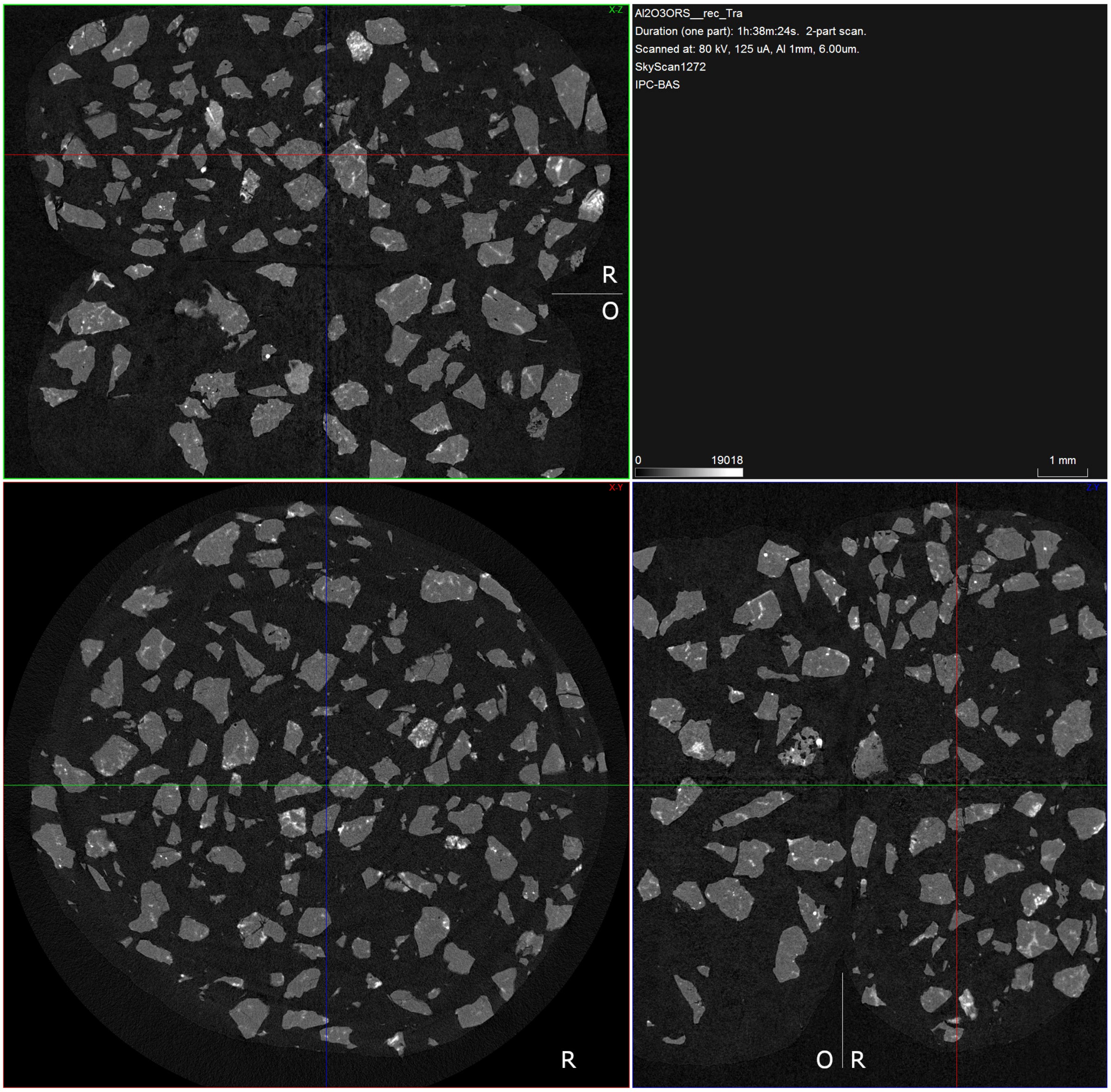

Fig. 5. Tomographic sections of the samples; sample R appears at the top in X-Z section, sample O at the bottom of X-Z section.

O are larger. Sample R contains both large and small particles. The large particles are inhomogeneous white, i.e. denser parts. According to the composition and the results of X-ray diffraction the most common phases are Al_2O_3, i.e. the particles, and iron oxides - the brighter spots. The iron oxides that are part of the abrasive are not a separate fraction of particles but are predominantly embedded in Al_2O_3. Some other impurities, denser than iron oxides, are also present but their volume fraction is much smaller and hardly matters for the abrasive properties. Sample O contains a little bit more impurities, which decrease after regeneration. The segmentation of the 3D images allows to conduct an individual analysis of each particle type and to determine particle size distributions and the volume fraction of iron oxides relative to the volume of Al_2O_3 particles. The results for volume fraction of iron oxides in Al_2O_3 particles is little, bigger for the original (2.76 %) vs. the regenerated Al_2O_3 (2.34 %). In addition, after the regeneration process, fewer impurities and pores remain - 0.08 % vs. 0.16 % in O-Al_2O_3. During the abrasive regeneration procedure part of the iron oxides and additional heavy impurities are lost. The volume fraction of iron oxides in one particle does not exceed 5 % for both samples.

The particle size distributions of the abrasives are shown in Fig. 6. Since the particles are irregular in shape, two quantities are used to represent the size distributions. The "diameter of a sphere with equivalent volume" is the diameter of a sphere that has the same volume as the real particle with an irregular shape. The second quantity is the maximum size of each of the particles. The distributions are different for the two samples. By diameter of a sphere with equivalent volume, the original specimen, O, has a narrow peak with a maximum at 0.8 mm and a wide base between 0.2 and 2 mm. After regeneration, this sharp peak disappears, the distribution is "blurred" and the maximum is moved to 0.6 mm. The effect of the abrasive crushing is obvious.

The character of the distributions of the maximum particle size is similar (Fig. 6, right), but the peak positions are of course larger than the diameter of the sphere with equivalent volume. It is worth to notice that the fragmentation of the recovered abrasive grains, accompanied by an expansion of the distribution of the abrasive particles in size is not due to heat treatment (pyrolysis) because at this temperature (600^{o}C) alumina oxide is stable. Obviously, these changes are due to the pressure (297 kg cm^{-2}) to which the discs are subjected during their production. Considering a publication in the literature Gomez, et al., it can be assumed that it is the expansion of the size distribution that has a negative effect on the performance of the abrasive [18].

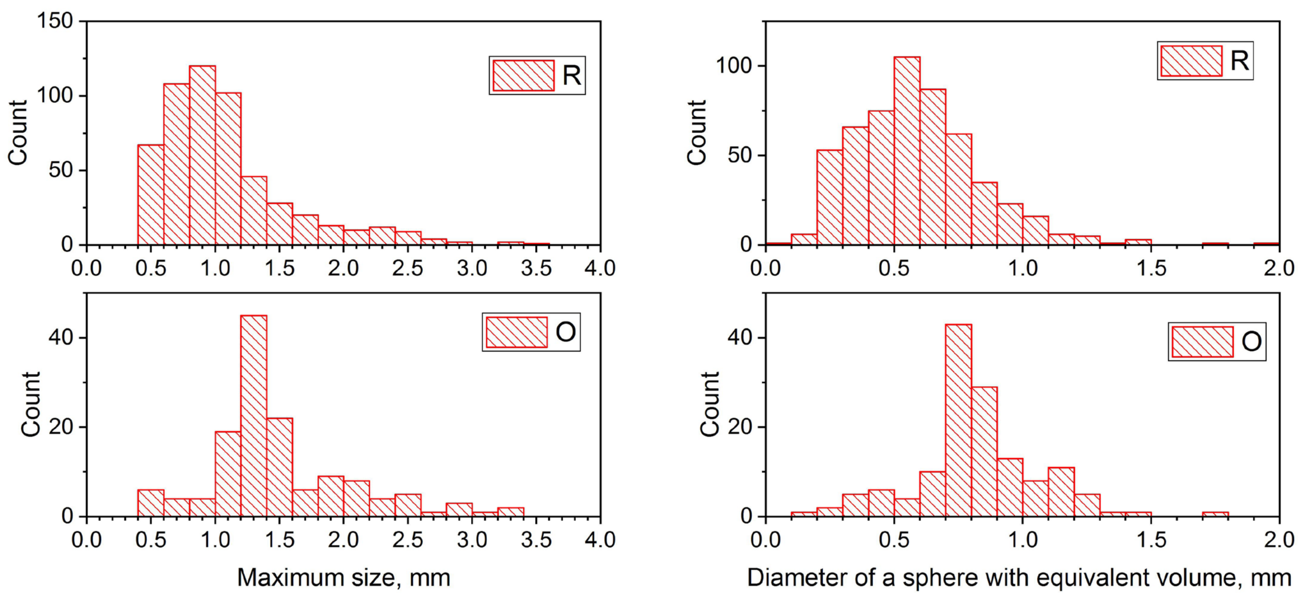

Fig. 6. Distribution of the particles by size: left - distribution of diameters of spherical particles with volume equal to the volume of the real particles; on the right - distribution of the largest size of the particles.

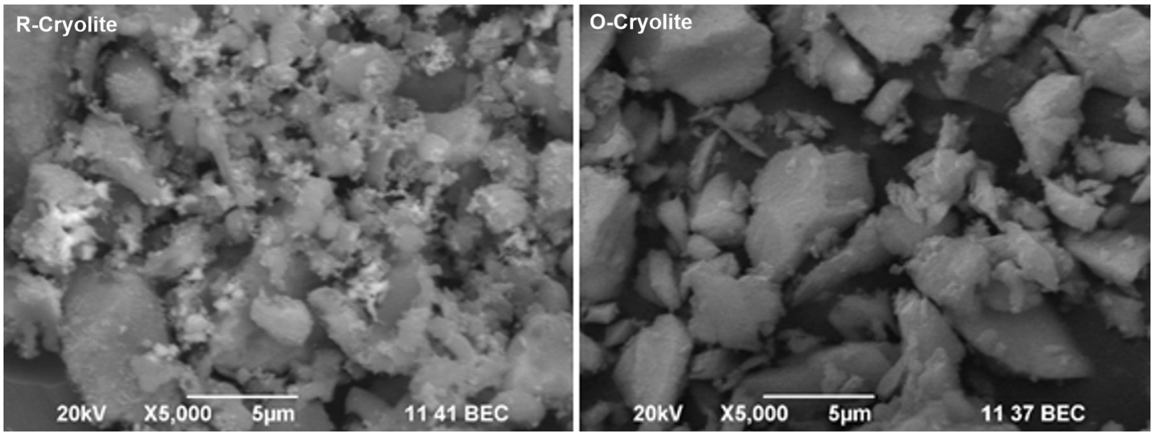

Fig. 7. SEM images of the recovered cryolite (left) and original cryolite (right).

Optical observation show that the fiberglass mesh does not change in size ot shape during the regeneration process. Within the error, all fibers have the same composition. Elemental analysis revealed that fiberglass contains O - 67.66 %; Al - 4.75 %; Si - 17.63 %; Ca - 7.89 % in the form of SiO_2, Al_2O_3 and CaO, respectively. Based on the tensile tests (1N) and the modulus of elasticity (14000 MPa), the recovered fiberglass weave can only be used to reinforce some fiberglass products without particularly high strength requirements, and only as medium layers. Fiberglass obtained from recycled sheet molding compound at 450^{o}C through this method has its strength lowered to roughly 50 % of that of new fiber. These fibers have been utilized as both a complete and partial substitute for new fibers in a dough molding compound. (DMC) [19].

Scanning electron microscope (SEM) studies (Fig. 7) of recovered cryolite revealed that there is a difference between recovered and virgin cryolite. After the pyrolysis process, the residue represents a mixture of Al_2O_3 and cryolite. The mixture was washed several times with water to remove the cryolite. The cryolite was precipitated from the wash water and then dried. The data from elemental analysis showed that in the recovered cryolite the content of carbon is 3 times higher compared to the original cryolite, i.g. the recovered cryolite contains charcoal, forming due to the pyrolysis of the phenol-formaldehyde resin. The cryolite is a filler, so carbon will not prevent it from being reused in the production of abrasive discs.

The recovered alumina was used for the preparation of cutting abrasive discs. Ten discs have been produced. Disc examination results are as follows. On a Speed test all disks can withstand an operating speed of 21193 RPM. The imbalance has a deviation of less than 1.5 %. The lateral pressure is less than 40 N. According to the quality standard of the cutting tests, a disc with a size of 125 mm diameter and 3 mm thickness should make 20 cuts, and the final diameter after these 20 cuts should be less than 110 mm. The discs we produced from the processed grain are 125 mm in diameter and 3 mm thick. After 20 cuts, the final diameter of the samples was 118 mm and 116 mm. The size and distribution of particles, as well as the shape of the abrasive particles, affect the cutting efficiency. These changes are due to the pressure (297 kg cm^{-2}) to which the alumina grains are subjected during their production. In our case, recovered alumina grains were twice subjected to the pressure.

CONCLUSIONS

A method was developed for the quantitative regeneration of Al_2O_3 from rejected and spent abrasive discs by pyrolysis, which overcomes the shortcomings of the method described in the literature, namely, the pyrolysis of the abrasive waste takes place without being preliminary crushed and the pyrolysis takes place at the same speed on the surface of the disc as in volume (thickness of the disk is ~ 3 mm). It was established that when the discs were placed vertically in the pyrolysis reactor pyrolysis proceeds at 600°C for 30 min and the organic bond is completely decomposed. In the temperature range of 600°C - 900°C the phenol formaldehyde resin decomposes while the abrasive material, the fiberglass mesh and the cryolite does not change. This method allows recovering of alumina, fiberglass mesh and cryolite.

The results from SEM images of regenerated and virgin Al_2O_3 show similar sharp edges. Elemental analysis reveals that the content of elements in the virgin Al_2O_3 and in the recovered one is the same. Angle distribution of recovered Al_2O_3 and virgin Al_2O_3 reveal that in both cases the maxima are between 80 and 120 degrees. The peak of the distribution of the particle size for recovered R- Al_2O_3 material are between 500 - 800 mm while for the virgin O-Al_2O_3 the dimensions are generally between 500 and 1000 mm.

The recovered alumina was used for the preparation of cutting abrasive discs. Disc examination results reveal that all disks can withstand an operating speed of more than 21100 rpm. The imbalance has a deviation of less than 1.5 %. After 20 cuts of the cutting test, the final diameter of the samples was 118 mm and 116 mm - according to standard it should be not less than 110 mm. The results obtained have a real contribution to the storage of raw materials, energy, labour and environmental protection.

Acknowledgments

This study was funded by the Abrasive Technology, Bercovitsa, Bulgaria. B.R. acknowledges the support by the European Regional Development Fund within the OP Science and Education for Smart Growth 2014-2020, Project CoE National center for mechatronics and clean technologies, No.BG05M2OP001-1.001-0008, and Project INFRAMAT-part of the Bulgarian National Roadmap for Scientific Infrastructure, supported by the Ministry of Education and Science

Authors' contributions*: D.T. conceived of the presented idea; K.T. wrote the manuscript with support from G.A.; I.Z., D. T., B.R. characterized samples with X-ray computed tomography and SEM. All authors provided critical feedback and helped shape the research, analysis and manuscript.*

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