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Obtaining Iron Nanoparticles from Chip Through Top Down Technology

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Abstract

The metal-mechanic industry has become one of the main economic activities in the world, however, it generates tons of iron shavings every year in the manufacture of parts and metal parts. This chip to be mixed with cutting fluids is waste and pollutes the environment.This paper presents results of recycled cast iron chip and its transformation in particles of iron in nanometersize obtained through the technology Top Down. The nanoparticles obtained have an iron content of 93.30% and low chromium content, a size in the range of 30 to 100 nanometers, these features allowed their application in the remediation of industrial wastewater contaminated.

Keywords: Chip; Recycled; Top down; Iron nanoparticles; Remediation

Introduction

The production or manufacture of parts and metal parts called machining process chip removal, consists of the transformation of metal materials into finished products, the material is ripped or cut with a tool giving place a waste or chip[1]. In this process there is a substantial loss of material in the metal cut, since the dimensions of the blank or ingot must correspond to measures greater than the piece to be manufactured. When comparing the weights of the finished piece and the blank, it can be seen that between 40 to 80% of the weight of the ingot is eliminated in chips depending on the shape of the piece[2]. The productivity of the transformation process by machining is directly related to the production of the chip, therefore, the greater the production of parts, the greater the production and volume of chips. Nowadays the demand of the market demands mass industrial production for which computerized numerical control (CNC) machines have been developed, which allow a greater production of pieces in a shorter time and therefore also the generation of greater volume of chip[3]. Another important factor to consider is that in the process of machining by chip removal, cutting fluids such as cutting oils or lubricant fluids are used for cooling and lubrication of the process, which mingle with the chip, if they are not treated by centrifugation or other methods normally contain 20 to 30% cutting fluid, so it can be considered hazardous waste if it reaches the ground or is dumped in landfills[4]. Of the total garbage generated, between 3 and 6% corresponds to metallic waste, among them the metallic shavings coming from the machining of parts[5]. A preliminary survey establishes that in the city of Sucre - Bolivia there are at least fifty metallurgical establishments that carry out this transformation with an average generation of 136 tons of metallic shavings per year (Figure 1a), making this survey extend to the national level reach 3500 tons (Figure 1b). Of the total of this chip 34% is cast iron (Torres-Espada unpublished data).

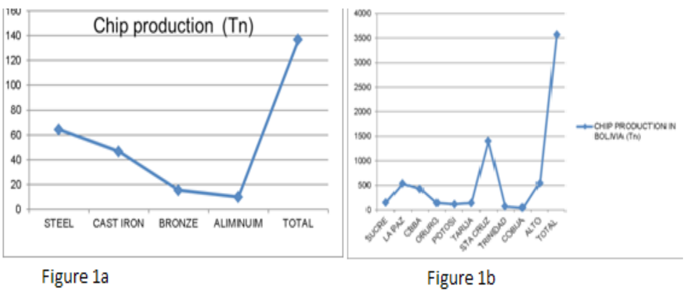

Figure 1: Production of metal shavings in Sucre and other cities in Bolivia until November of 2017.

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In other areas of pollution according to[6]" The most polluted waters of mining in Bolivia are in the San José mine (Oruro) has a pH of 1.0 to 1.2 and a concentration of relatively high chemical elements of lead, arsenic and heavy metals as well as sulfates, chlorides and sodium ".This scenario is repeated in many other mining centers in the country, many of which are in population centers, which affects the ecosystem and the health of the surrounding population due to exposure to lead[7]. On the other hand, levels as high as 80 ppm of chromium have been observed in the waters and soils of industrial zones[8], due to the inadequate waste disposal and leaks of industries such as paint and pigment, refractory, tanning of leather, chrome plated, textiles, preservation of wood, among others. One of the most contaminating heavy metals is chromium, whose most stable states in most natural environments are (+ III) and (+ VI) (Cotton, 1999); however, hexavalent chromium is the most toxic form for humans, animals, plants and microorganisms, because it has a significant solubility and mobility[8].

Therefore, it is urgent to remediate these contaminated waters by accessible and economical methods, before dumping them into the rivers, in order to avoid greater contamination, one of the alternatives being the use of iron nanoparticles.

The problem identified as the reason for the present investigation suggests that; the cast iron chip from the machining process is a waste and pollutes the environment but could be valued by converting it into a potential source for the generation of nanoparticles for environmental remediation. Being the recycled of the cast iron chip very important for the reduction of the environmental pollution, we propose to produce iron nanoparticles through the top-down method with size characteristics, physical and chemical properties that allow its economic and competitive application, in biotechnology, especially in the field of remediation of contaminated mining and industrial waters in Bolivia. In the present investigation, the recycling of cast iron shavings was carried out using Top-Down technology, obtaining high-value-added iron nanoparticles for different applications in nanotechnology, especially in the remediation of contaminated water and soil, as well as processing obtain by-products for various applications. There were trials and tests appropriate for the characterization and verification of the quality of the obtained nanoparticles. The cost-benefit efficiency of the method in the production of nanoparticles was evaluated.

Finally, the experimental treatment was carried out with the iron nanoparticles obtained from model solutions for the remediation of wastewater contaminated with chromium, from a tannery.

Method and materials

The methodology applied to obtain iron Nano powder was Top - Down technology by mechanical method using a High Energy Ball Mill (HEBM) and subsequent selection and magnetic separation, as it is the most economical, simple and accessible. The cast iron chip was collected from metalworking and local grinding companies. The size and shape of the cast iron chip is very varied, its main characteristic is to be fractionated and discontinuous in sizes between 1 to 15 mm, and torsion angles from 0 to 180 degrees with a fragile consistency.The preparation of the chip consisted of making a meticulous selection to avoid the presence of foreign materials for which magnets were used to select only the metal. On the other hand, separated steel and non-ferrous metal chips. Once the cast iron chip was classified, the mill was grinded for which a shot mill was used, which was specially constructed for this purpose. Characteristics of the mill (figure 2): outer length: 220 mm, outer diameter: 170 mm, drive with gear motor of 0.25 kW, reducer speed ratio 1350 to 150 rpm, weight of pellets 3 kg of pellets, diameter of grinding bodies: 25 mm.

Figure 2: Grinding of chip of iron cast in a pellet mill / (source: own production)(Unpublished photo)

The test of the mill with different charging regimes was determined that the proper weight of load to get a fine grind is 1000 g with an average time of 18.18 minutes. The selection of the size of particles of iron was made by sieving of the product in calibrated sieves which were acquired in www.coleparmer.com/Virtual-Catalog/es-us/1273 their characteristics are:

Material: steel stainless type, set of sieves U.S. standard mesh size: each one in the following screen sizes: #5 (4000 µm), #10 (2000 µm), #35 (500 µm), #60 (250 µm), #125 (µm) #230 (63 µm), #325 (45 µm) height: 2 (5 cm) (inch)

For this stage was built a vibrating machine of the following features: drive with gear motor power: 1.1 kW; Speed: 2800 rpm; Sieving stages: 6

Figure 3: Machine vibrating sieve for the selection of the size of particles of iron.(source: own production)(Unpublished photo).

Stages of product selection ground:Ground product was dosed to the thickest shale #5 of 4000 microns through mesh 60 MESH, in this shale was obtained a 250 Micron powder; It was ground again to get to the next measure of 230 Micron with a net 63. With a sequence of tests performed in the vibrating machine sieve shaker was determined that the yield reached is 0.201 Kg/h. The fine dust extraction was done in the last stage of the sieved through mesh 325 mesh hole 45 µm, which are also particles of nanometer size.

Collection of nanoparticles of iron nanoparticles:
Collection was performed by two procedures:
(a) Through a liquid solution of iron particles more alcohol isopropyl with magnetic field following these steps:[9]

I. After the selection process, the Nano powders of iron were introduced in a Cuba with isopropyl alcohol dispersed using ultrasonic agitation.
II. With a permanent magnet placed above the surface of the container containing a solution of (particle + isopropyl alcohol) is attracted to all the particles to the surface of the solution.
III. Complete sedimentation of particles and because of the weight, the finest particles were the latest to fall.
IV. Reapplying the magnet at one distance greater than 4 to 10 cm to attract children.
V. Once removed the magnet, nanoparticles can be collected using a syringe 10 ml neojet.
VI. Finally, the solution (nanoparticles + isopropyl alcohol) mixed in be deposited in another tank with magnetic wallpaper to spread the spirit of nanoparticles, which were then dried in an oven at 200°C.

(b)By via dry:
For this procedure is built a high energy ball mill (HEBM) of the following characteristics:

Body of the mill diameter 300 mm, height of the body of the mill 400 mm Rotary shaft with 16 blade, grinders diameter bodies: 35, 25- and 10-mm drive by means of a drill press with engine of 1.5 cv, vacuum cleaner with filter 3 M N95.Collection was performed with the mill and cleaner running at intervals of 30 minutes.

Figure 4: Procedure for collection of nanoparticles of iron through the dry mill of high energy (Source: own production)(Unpublished photo)

Determination of the composition of nanoparticles of iron:
The determination of the chemical composition of iron nanoparticles is carried out following the experimental method. The objective of this test was to determine the percentage of iron and chromium in samples of iron nanoparticles, as well as other traces. Components present in the samples of iron nanoparticles were determined using the method of mass spectrometry of dispersion of energy of x-rays (EDS) on an equipment of the following features: portable X-ray Fluorescence X Name Team: X-RAYFLUORECENCE SPECTROMETER, mark ELVAX model: CEP-01AAEC412131001 brand Prospectors LE. To perform elemental analysis in X-ray fluorescence equipment, must have porta samples which must be covered by a film on the bottom, this is good practice, due to the fact that this film does not affect the time of reading of the sample. The sample must be homogeneous and cover more than 50% of the slide shows. The beam of light must affect in the center of the slide samples. Once the equipment was turned on, the sample was introduced inside it to later identify the elements that are observed on the equipment screen. The statistical treatment was performed for measurements n = 29, degrees of freedom = 28 and a significance of $\alpha = 0.05$ for an iron nanoparticle. Finally, once the measurement proceeded to record the data of the identified elements that in this case only took into account the iron (Fe) and chromium (Cr).

Determination of the size of the nanoparticles of iron:
a) The test was performed on an equipment of the following characteristics: scanning electron microscope; Scanning Electron Microscope (SEM), brand of measuring instruments: JEOL (field emission cannon JSM 7900f), increases capacity: 500 X 200000 x enlargement of the image. Detector: Upper Electron Detector (UED); stress of acceleration i.e. 1.00 kV Mode Gb = Electron type
The experimental procedure followed this process consisted of:

- Dust deposition observed in small Silicon plates covered with gold.
- Removal of lumps and excess dust by blowing air with a pipette
- Metallization of powders with 15 nm of gold to repair them and prevent the contamination of the microscope camera.

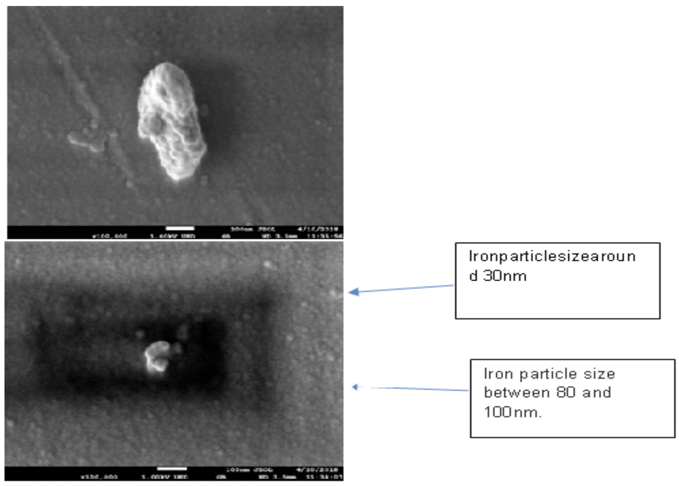

Figure 5: Image of iron nanoparticles observed with 100,000 X magnification
Source: Lab. Nanomaterials Uni. La Salle - France)(Unpublished photo)

Results

Nanoparticles of iron were obtained using the Top-Down technology from the manufacturing process-from chip.

In the grinding process determined that the optimal weight of load to the mill was of 1000 g with a time of 18.18 minutes, with which it could be determined that the performance of the grind is 3.3 kg/h.
In the machine sieve with mesh 325 45-micron MESH obtained

powder of iron with a yield of 0.2 kg/h.

At the stage of collection through a liquid of nanoparticles of iron in solution with alcohol isopropyl is achieved by magnetic separation from 45 µ iron powder weighing 210 grams of nanoparticles iron with a yield of 0.11 kg/h.

In the stage of collecting dry managed to get 45 grams of iron nanoparticles by filtering in two hours, with a yield of 0.023 kg/h.

This result shows that the process of obtaining via liquid is more efficient.

Components present in the samples of nanoparticles were determined using the method of x-rays (EDS) energy dispersive spectrometry. The results are detailed in table 1.

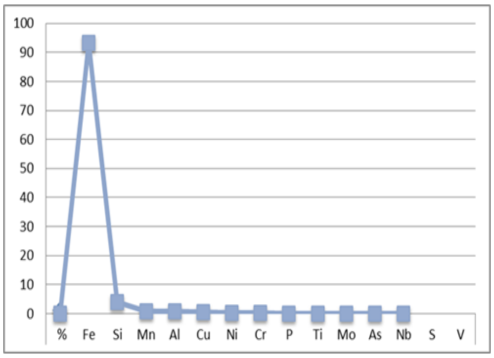

Figure 6: Average chemical composition of iron nanoparticles obtained using the method EDS (Source: own production) (Unpublished photo).

The size of nanoparticles in a first test was determined by measurement of captured images in the scanning electronic microscope with an increase in 100 X, noting a size minimum of 778 nm with irregular shape. Nanoparticles with 30, 80 and 100 nanometers with irregularly shaped carving were observed in the second test in a scanning electron microscope (SEM) of 200000 X increase. In the work of research thesis entitled "experimental treatment of water polluted by chromium (VI) through the use of iron particles obtained from recycling of chip technology top down"[10], applied the nanoparticles of Iron obtained, for the remediation of contaminated water with chromium, the results were as follows:

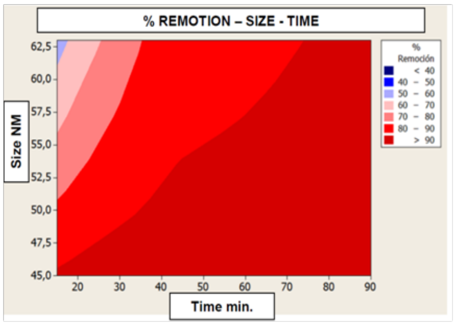

Figure 7: Graphic of contour removal (%) vs. size (nm) and time (min) (Source: P.Conde . J. Torres 2018) (Unpublished photo)

In Figure 7, you can see that it can reach high percentages of removal of chrome depending on the size of nanoparticles; how much smaller the size of nanoparticles is can reach high percentages of removal from the first 15 minutes. The final conclusions of the research are as follows:

I. The determination of the percentage of removal of chromium (VI) in the tests resulted in that all races reached approximately the same percentage of removal at the end of 90 minutes, which corresponds to one value greater than 90%. (Fig. 8)

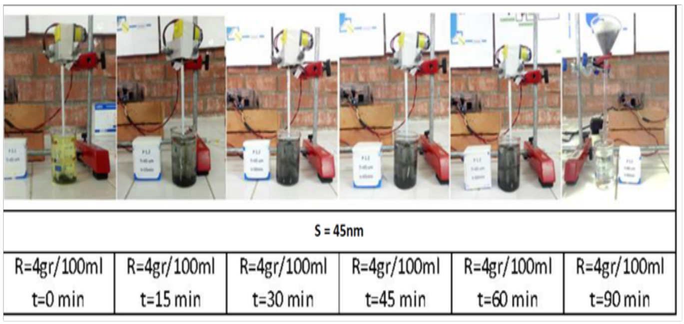

Figure 8: Evolution of the water sample treated in time[11](Unpublished photo)

Table 1: Determination of components of the sample of nanoparticles of iron using the EDS sample of iron nanoparticles. (Source: P.Conde . JSTorres 2018).

%	S1	S2	S3	Average	Variance	Desv. Standard	% coefficient of variation	Confidence interval	Reading
Fe	93,17	93,22	93,5	93,30	0,03163	0,17786	0,191	0,4	93,30 ± 0,4
Si	4,01	3,81	3,84	3,89	0,01163	0,10786	2,775	0,3	3,89 ± 0,3
Mn	0,80	0,84	0,79	0,81	0,00070	0,02646	3,266	0,07	0,81 ± 0,07
Al	0,77	0,8	0,77	0,78	0,00030	0,01732	2,221	0,04	0,78 ± 0,04
Cu	0,55	0,6	0,46	0,54	0,00503	0,07095	13,220	0,18	0,54 ± 0,18
Ni	0,29	0,32	0,32	0,31	0,00030	0,01732	5,587	0,04	0,31 ± 0,04
Cr	0,19	0,18	0,19	0,19	0,00003	0,00577	3,093	0,014	0,19 ± 0,014
P	0,07	0,11	-	0,09	0,00080	0,02828	31,427	0,07	0,09 ± 0,07
Ti	0,07	0,05	0,08	0,07	0,00023	0,01528	22,913	0,04	0,07 ± 0,04
Mo	0,04	0,04	0,03	0,04	0,00003	0,00577	15,746	0,014	0,04 ± 0,014
As	0,02	0,01	0,02	0,02	0,00003	0,00577	34,641	0,014	0,02 ± 0,014
Nb	0,01	0,01	-	0,01	0,00000	0,00000	0,000	0,00	0,01 ± 0,00
S	-	-	-		-	-	-	-	-
V	-	-	-		-	-	-	-	-

II. You can tell that the use of iron nanoparticles obtained by recycling chip technology top down is a good method for removing hexavalent chromium, since it uses an economic reducing agent that can be easily deployed to any industry which found hexavalent chromium in its wastewater.

Discussion

Table 1 determination of components of the sample of nanoparticles of iron using the EDS, show that the variance in measurements and the standard deviation of the measures are quite low. By which it can be concluded that the measurements for the determination of the chemical composition of iron nanoparticles are extremely precise.

The results of experimental treatment of water polluted by chromium (VI) through the use of iron particles obtained indicate that the obtained nanoparticles have an iron content of 93.30% and low-chrome, with a size that is in the range between 30 and 100 nanometers, allow its implementation in the field for the remediation of industrial waste water contaminated with chromium. In the article "Iron nanoparticles produced by high energy ball milling"[10], to obtain the iron nanoparticles, a metallic powder with a cubic structure centered on the faces with purity of 99.9% acquired in Alfa-Aesar Co. Instead, in the present project, the source material is metal shavings of cast iron from the machining of parts, considered as polluting waste, constituting a potential reso/rce for obtaining iron nanoparticles with an important impact on the protection of the environment by recycling and its application.

The use of iron nanomaterials has received a lot of attention due to its unique property, such as high ratio of surface area to volume, modifiability of the surface, excellent magnetic properties and great biocompatibility. Iron nanomaterials are efficient Nano sorbents for heavy metals and organic pollutants are the most attractive and successful applications[12]. For this reason, the present investigation shows that iron shavings constitute a potential resource for obtaining iron nanoparticles with a significant impact on the protection of the environment.

Analysis of production cost of nanoparticles of iron from[13-16] cast iron chip is evidence that it is very convenient in terms of cost-benefit in comparison to the imported product since it reaches to 52% less than the price International ($86 / 100g). With the present research it is demonstrated that the method used for the recycling of the cast iron chip for the obtaining[17-22] of iron nanoparticles with top down technology is the simplest and accessible.

On the other hand, with the production of nanoparticles of iron from cast iron chip[23-29], it is possible to reduce the amount of chip at the departmental level[30] in 34%.

Conclusions

This research helped obtain iron nanoparticles with size between 30 and 100 nanometers and purity of 93.30% using Top-Down technology with equipment designed and built specifically for this purpose, the methodology developed allows a competitive and feasible production of scaling large scale with very affordable costs.The contribution of the research lies mainly in the obtaining of nanoparticles of iron of multiple applications, especially on remediation of water and contaminated soils, which are found in the mining, industrial and petroleum sector in Bolivia.
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