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Chemical Extractive Technique for Commercial Purity Metals

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Abstract

Commercial purity iron powders were produced by using a new hydrometallurgy process. It was found that the most important factor in enhancing the purity of iron was the number of water washing process. X-ray diffraction pattern showed that the high peak purity of iron powder increased with increasing the number of water washing. The developed new methodology was based on the reaction between the aqueous ferrous sulfate and the hydrochloric acid with the presence of high purity aluminum flake. The purity of iron powders increased considerably with increasing the multi-water washing for leachate containing iron powders. The purity of iron powders was reached up to approximately 93.5%. The mean particle size distribution and apparent density for the highest value of purity are 50-100 µm and 2.85 g/cm^3 respectively.

Keywords: Commercial iron powder; Hydrometallurgy process; Iron sulfate; Hydrochloric acid; Hydrometallurgy

Introduction

Although many metals can be produced in the form of powders with high purity, only a few are used in the manufacturing of bulk metals or alloys. On the other hand, powder technology is well recognized as an important primary manufacturing process consists of many steps [1]. The most important step in any powder technology is the manufacturing of powder using huge numbers of mechanical, physical, chemical, electrochemical, thermal processes [2]. Hundred thousand tons consumption of iron and steel powders has been increasing considerably worldwide [3]. The quantity and quality of iron powders are governed mostly by the route of manufacturing. The high-quality iron powders with considerable quantity are produced by many chemical routes from many aqueous ferrous solutions [4]. The iron powders produced by such route have important properties of low oxides, uniform particle shapes, good sinterability, and high apparent density [5].

The overview of iron powders produced from chemical routes depends on the type of the chemical process. The open literature, in the production of iron powder from the technique of solution and decomposition from their compounds, is advanced well [6]. However, the aqueous metallurgy or mechano-chemical processes are relatively new technique. Sulfates are widely accepted as important inorganic materials having many scientific and industrial applications [7]. The most important applications of iron sulfate are soil, animals, and humans for plants, the reducing agent, reducing toxicity in cement, dye fixative in the textile industry and blacken the leather [8]. There are many application of iron oxide powder as a coloring and coating material, iron oxide as the catalyst, as the gas sensing material, as impurity control agent, as electromagnetic material [9]. The important thing for preparation of nanoparticles is the consisting of pure iron a complicated task because they always contain oxides, carbides and other impurities [10]. Magnetic nanoparticles of iron oxides (Fe_3O_4, γ-Fe_2O_3) have attracted attention in biomedical applications like drug delivery systems, magnetic resonance imaging, and cancer therapy, but also as adsorbents in water purification due to their numerous advantages [11]. Magnetic iron oxide nanoparticles have also displayed a large number of biomedical applications; the most common application is considered in Magnetic Resonance Imaging (MRI) as contrast agents [12]. Various synthetic methods are continually being improved, uniform α-Fe_2O_3 particles within the nanometer range (100–300 nm) have been obtained by precipitation of iron (III) perchlorate in the presence of urea [13,14]. Electrodeposition process refers to a film growth process, which consists in the formation of a metallic coating or powders onto a base material occurring through the electrochemical reduction of metal ions from an electrolyte [15]. Metal powders can be produced by electrodeposition from aqueous solutions and fused salts [16]. This method is reversed adaption of electroplating. Hard and brittle mass are deposited. They are subsequently ground to powders having mainly dendritic shapes [17]. The electrodeposition process consists essentially of the immersion of the object in a vessel containing the electrolyte and two electrodes. It followed by the connection of these electrodes to an external power supply to make current flow possible [18]. The workpieces are connected with the negative terminal of the power supply. In such a way that the metal ions are reduced to metal atoms. they eventually form the deposit on the surface. Fig.2.1 shows the schematic diagram of electrodeposition methods [19].

It is used extensively in the preparation of iron, beryllium, copper and nickel powders. Adjustment of the chemical and physical conditions during electrodeposition makes it possible to cause the metal to deposit loosely on the cathode of the cell either as a light cake or in flake form. Both are readily crushed to a powder. The method yields a high purity metal with excellent properties for conventional powder metallurgy processing. The process involves the control and manipulation of many variables and in some cases is significantly more costly than other techniques [19,20]. There are two practical methods of obtaining powder by electrodeposition direct deposition and brittle cathode process [20].Direct deposition of a loosely adhering powder or spongy deposit that can easily be disintegrated mechanically into fine particles. Direct deposition of powder or sponge on the cathode is achieved by controlling the composition, temperature, rate of circulation of the electrolyte, current density, size and type of anode, cathode and their distance from each other, quantity and type of addition agent and removal of deposits at the cathode brush-down interval. The shape of electrolytically powder particles obtained depends on the metal deposited and the operating conditions. Electrolytic copper deposited from a sulfate /sulfuric acid electrode forms dendritic particles [14,20].

Material and experimental procedures

In this study, the iron sulfate ($FeSO_4.7H_2O$) was synthesized by chemical route. It has been produced by the reaction of low carbon mild steel scarp chips with H_2SO_4. High quantities of wet iron sulfate were produced. This iron sulfate was dried at 110 ºC to obtain partially dried iron sulfate with green color. Chemical analyses of iron powder were determined by using electro spark analysis after compact iron powder as round bulk in the table (2). The prepared iron sulfate was ground into particle size less than 100 µm. Also, the flake of pure aluminum (particle size less than 250 µm) as catalyst and 36.5% concentration Hydrochloric acid as reaction agent have been used in this study. This process was selected due to the low cost and availability of starting materials.

Thousand gram of iron sulfate and 50 g of high purity aluminum flake were mixed thoroughly and then placed inside a glass vessel. The hydrochloric acid of 250 cm^3 was added to the mixture. The reaction process was carried out on the stirrer at 250 rpm for a period of 30 min at room temperature. At the end of the reaction, a standard Buchner funnel and the vacuum pump were applied to filtrate the reactant. The outcome of the reactant was washed many times. The process of washing the reactant was taken place by immersing the reactant in 1000 cm^3 of distilled water. The mixture is thoroughly washed for a period of 15 min by the string at 250 rpm. At the end of each water washing, the reactant was dried and new water was added to the same procedures. Different numbers of washing were carried out to determine the purity of iron. At the end of each washing, a 10 g sample was collected and dried under argon gas at 37 ºC for 90 min to prevent oxidation. The iron particles after drying were separated by magnetic. Fig. (1) represents the Flow Chart of Experimental Work.

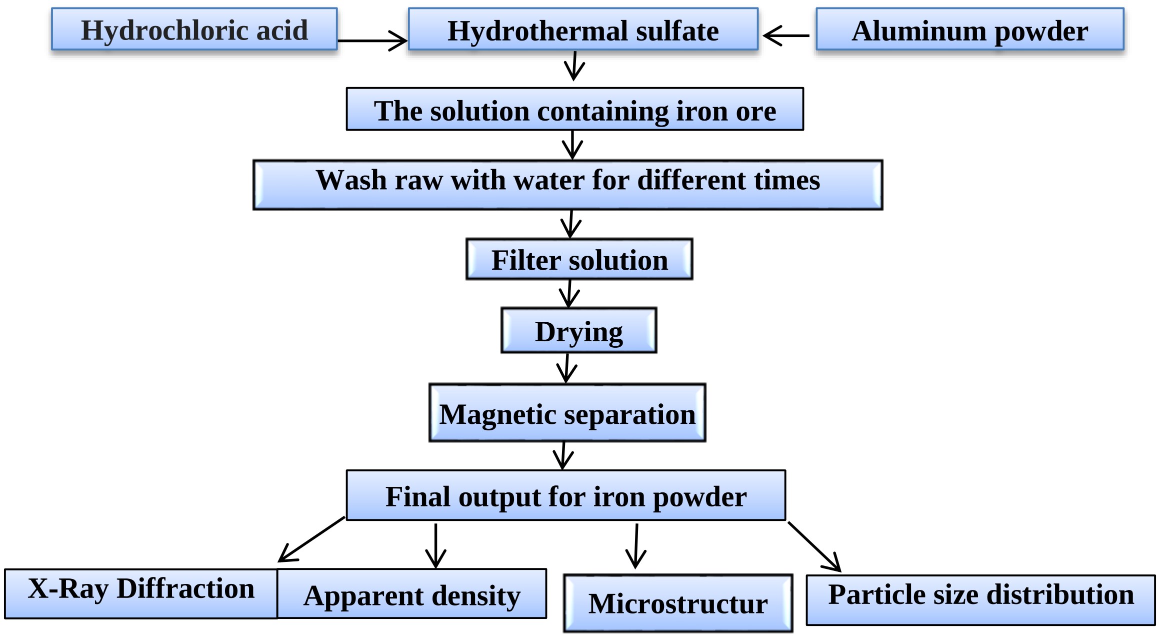


Figure (1): Flow Chart of Experimental Work

Results and Discussion

Chemical analysis of the leachates before magnetic separation confirmed the chemical reaction of iron sulfate and hydrochloric acid at the presence of Al flake as below:

$$3FeSO_4.7H_2O + 3HCl + 3Al \longrightarrow 3Fe + Al_2(SO_4)_3 + AlCl_3 + 7H_2 \quad …….. (1)$$

It was found that the role of Al flake in the presence of HCl is to reduce iron sulfate to iron with different purity dependent on the number of washing. Table (1) shows the particle size distribution (PSD) of the iron separated magnetically at different washing numbers; there are within narrow range of 50 to 100 µm. figure (2) and (3) shows the iron powder after magnetic separation with different particle size distributions and volume percentages. figure (4) and (5) shows the shape and SEM microstructure of the iron powder, the SEM image shows a spongy and some dendritic morphology that present e due to the realest of hydrogen gases in the Interaction result.

Table (1): Weight % of iron at different particle size distribution for different washing number.

Washing Number	%50 µm size,	%100µm size,	%150µm size,	Total % for all size
3	29	39	32	100
6	32	40	28	100
9	40	36	24	100
12	51	32	17	100
15	55	30	15	100

The vital results are that with increasing the washing number the percentage of a lower size of particles increased considerably. This is believed to be due to leaching of impurities from the iron sulfate. Table (2) represents the high purity of iron particles at different washing numbers. Figure (6) shows the X-Ray diffraction pattern for the iron powder that shows all strongest peak for iron. Figure (7) shows clearly that the increase of the washing numbers enhanced highly the purity of iron. This result confirmed that the small size of particles improved the layer charges sensitively to water soaking. Increasing the number of water washing increased the electrical field generated from the charges of iron sulfate and that results from flow the equation ($y = -0.4167x^2 + 10.917x + 22.8$) where the y-axis represents the Purity of iron, wt % and the X-axis Water washing number. Reducing the electrostatic attraction between the components of iron sulfate led to increasing the purity and removal the contaminants. This is clearly seen from Figure (8) where the weight gain of iron reduced considerably with increasing the water washing number and that results flow the equation ($y = 0.2143x2 - 6.2238x + 98.5$) where the y-axis represents the Iron gain, g of iron, wt % and the X-axis Water washing number.

The interesting data gathered from apparent density of iron powders are the apparent density increased with increasing the purity of iron Figure (9) and that results flow the equation ($y = 0.2601\ln(x) + 2.1672$) where the y-axis represents the Iron gain, g of iron, wt % and the X-axis Water washing number. This is contradicting to the fact that with reducing the particle size distribution the apparent density reduces. The main reason is believed to the supporting data obtained that enhancing the purity of iron and removal of the contamination reduce the friction between the particles.

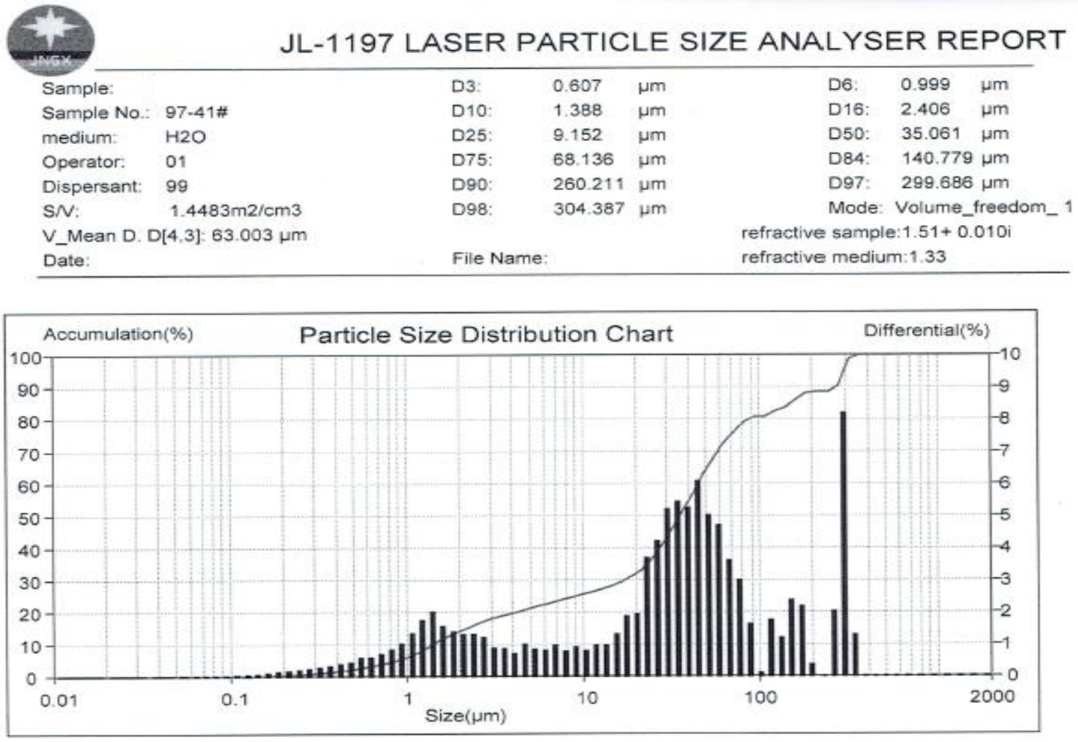


Figure (2): The particle size distribution for iron powder the mean particle size equal to 50-100

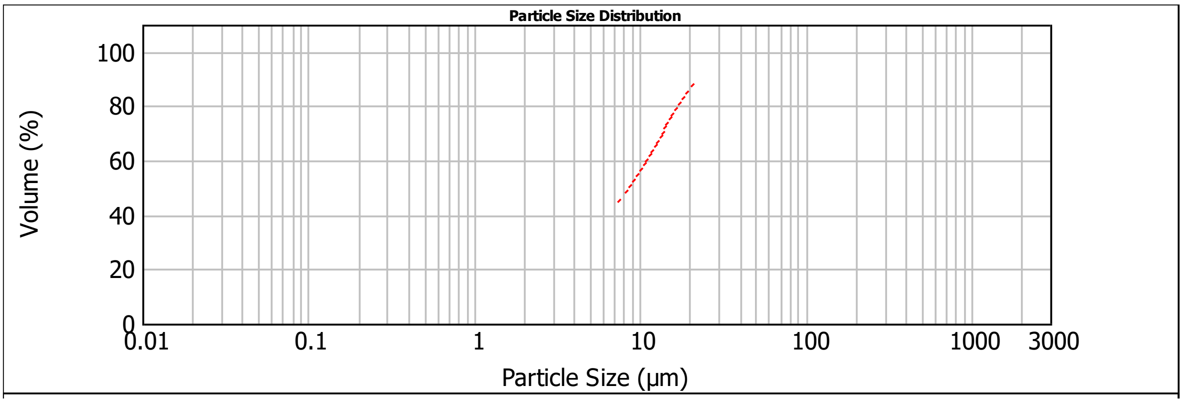


Figure (3) the mean particle size distribution for iron powder and the high volume percentage in the rang 50-100 µm.

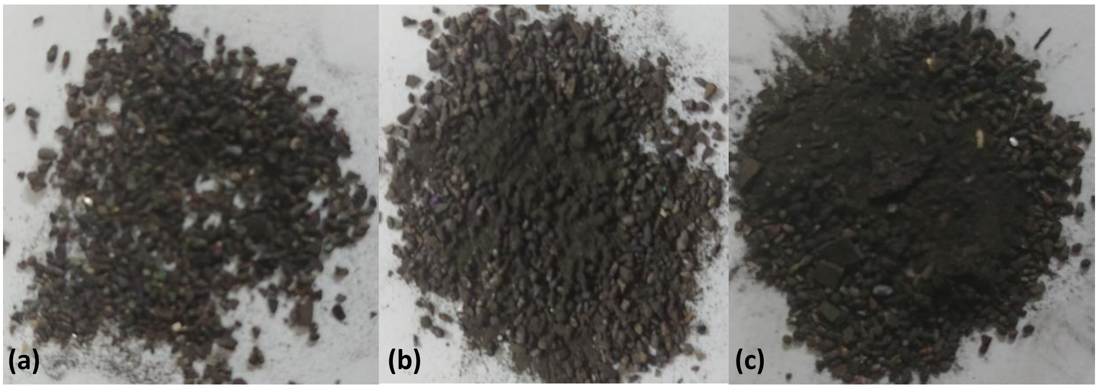
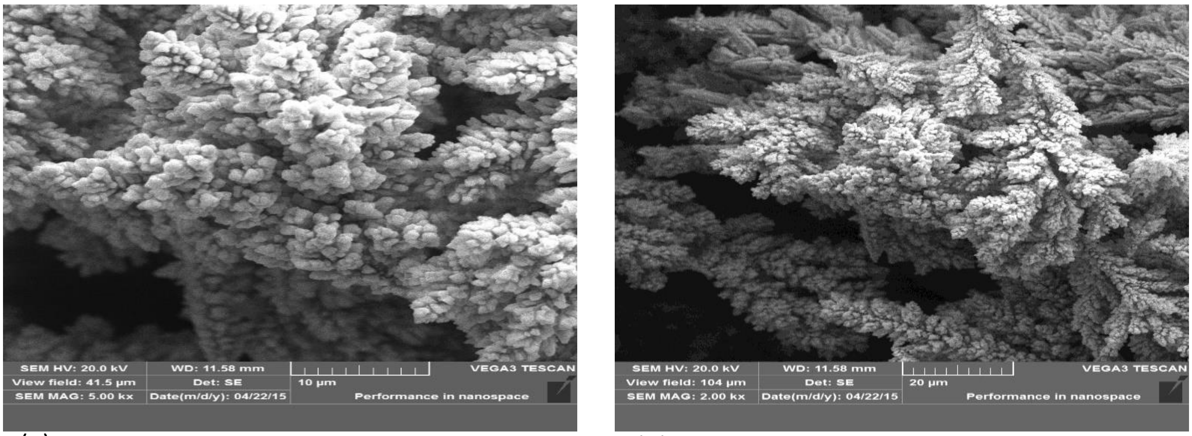


Figure (4): Shows (a, b and c) with different particle size of iron powder after magnetic separation.



(a) (b)

Figure (5): Shows the SEM images (a & b) spongey and dendritic morphology due to release hydrogen in the reaction for the iron powder that produced.

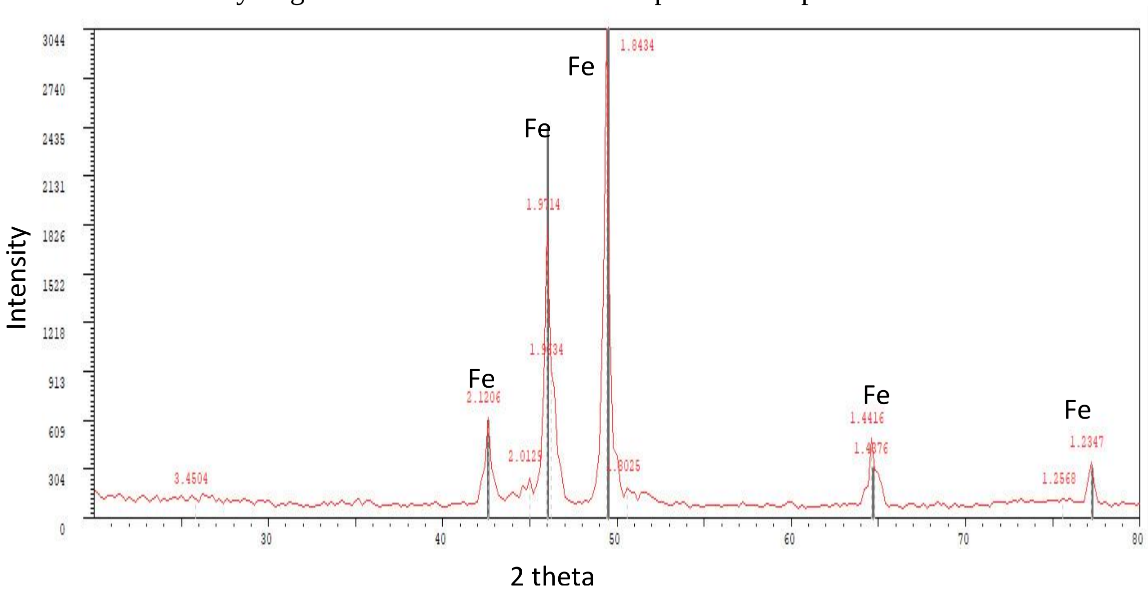


Figure (6): The X-Ray diffraction pattern for the iron powder that shows all strongest peak for iron.

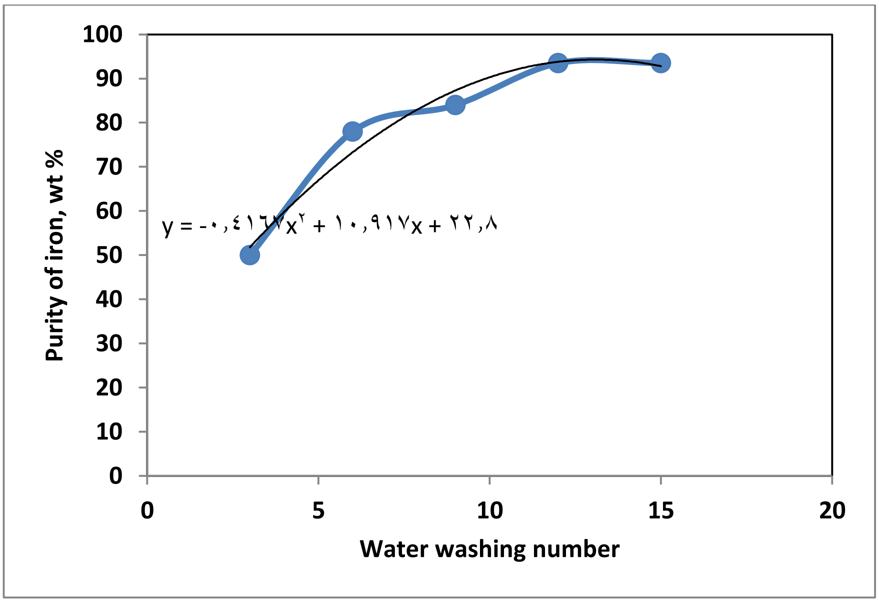


Figure (7): The relationship between water washing number and the purity of iron.

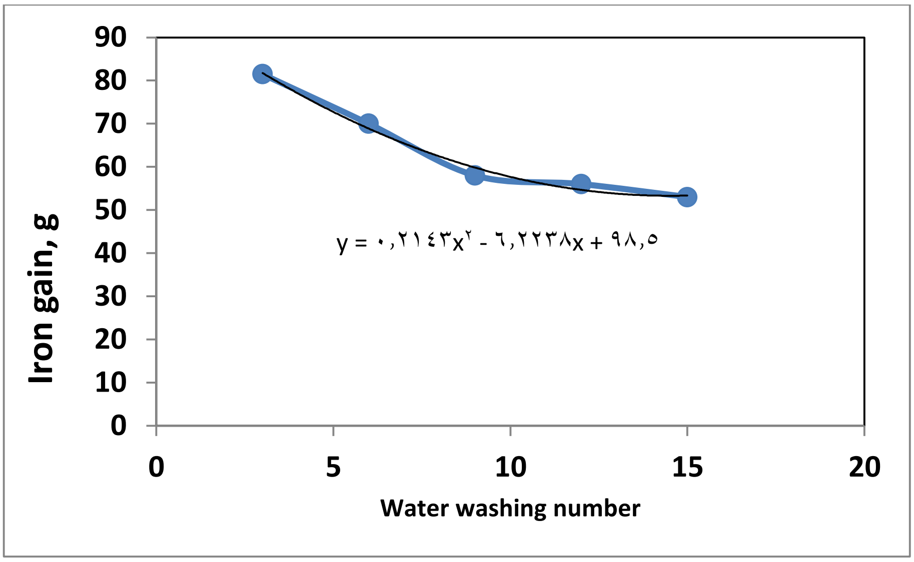


Figure (8): The relationship between the water washing number and the amount of iron obtained.

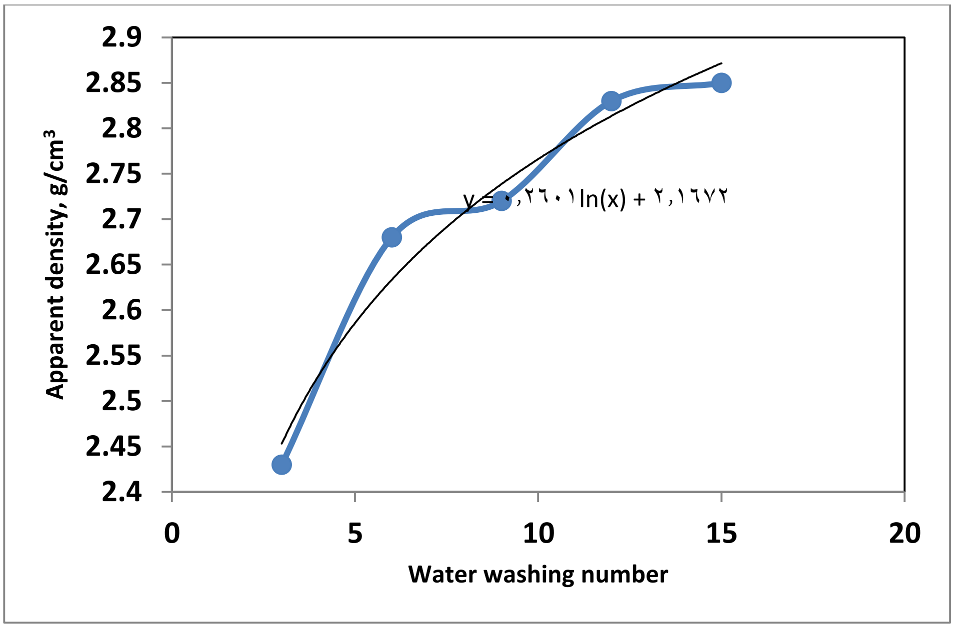


Figure (9) The effect of iron purity on the apparent density.

Conclusions

1- It has been shown that the number of washing increases the purity of iron considerably.
2- The apparent density of iron increased noticeably with increasing the purity of iron.
3- Lower temperature and prolonged drying should be achieved in order to prevent oxidation of the iron powders.

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تقنية الاستخلاص الكيميائي للمعادن ذات النقاوة التجارية

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الملخص

تم انتاج مساحيق من الحديد باستخدام تقنية جديدة بالطريقة الميتالورجيا المائية. لقد وجد بان اهم عامل يؤدي الى تحسين نقاوة الحديد هو عدد مرات الغسل بالماء. اوضح التحليل الدقيق باستخدام نموذج حيود الاشعة السينية بين زيادة نقاوة محتوى الحديد مع زيادة عدد مرات الغسل بالماء. ان الاساس بخطوات العمل بالطريقة الجديدة المطورة هو التفاعل بين كبريتات الحديد وحامض الهيدروكلوريك بوجود رقائق الالمنيوم عالي النقاوة. ازدادت نقاوة مسحوق الحديد بصورة ملحوظة مع زيادة عدد مرات الغسل للعصارة الحاوية على مساحيق الحديد. ازدادت نقاوة مساحيق الحديد الى حوالي 93.5%. أن توزيع حجم الدقائق والكثافة الظاهرية عند اعلى قيم للنقاوة هو 50-100 مايكرون و 2.85 غم/سم^3 على التوالي.