# CHARACTERIZATION OF DIFFERENT ELECTROLYTIC IRON POWDERS

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# CHARAKTERIZÁCIA ELEKTROLYTICKÝCH PRÁŠKOV ŽELEZA PRIPRAVENÝCH RÔZNYM SPÔSOBOM

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**Abstrakt**

Práca hodnotí sedem vzoriek elektrolytického práškového železa, ktoré sa pripravili zo síranu železnatého, odpadu z Egyptian Iron and Steel Company. K objasneniu primárnych a sekundárnych charakteristík týchto práškov bola urobená sitová analýza, zmeraná ich hustota a produkt bol študovaný pomocou skanovacieho elektrónového mikroskopu (SEM) a svetelného mikroskopu. Primárne charakteristiky zahrňujú: tvar častíc, povrchovú textúru, veľkosť častíc a mikroštruktúru, zatiaľ čo sekundárne charakteristiky: zdanlivú hustohu a hustotu prášku po strasení, vzájomné trenie medzi časticami, tekutosť práškov, ich kompresibilita a spekateľnosť.

V práci bol ďalej študovaný vplyv primárnych charakteristík na sekundárne charakteristiky. Častice železa v rôznych vzorkách boli mierne porézne a mali polykryštalický vzhľad. Prášky tvorené časticami s členitým povrchom vykazovali vyššiu zdanlivú hustotu pri vyššej priemernej veľkosti častíc, zatiaľ čo prášky tvorené časticami s relatívne hladkým povrchom vykazovali nižšiu zdanlivú hustotu. Hustota surového výlisku, parameter zhutnenia a stupeň spekania v prípade jemných práškov bol vyšší ako u spekaných hrubozrnných práškov.

**ABSTRACT**

The present investigation was carried out on seven samples of electrolytic iron powders prepared from ferrous sulphate by-product which is one of the wastes of the Egyptian Iron and Steel Company. Sieve size analysis, scanning electron microscope, reflected light microscope and densities measurements were used to elucidate both the primary and secondary characteristics of the different powders. The primary characteristics included particle shape, surface texture, size and microstructure, while the secondary characteristics included apparent and tap densities, interparticle friction, flow time, compressibility and sinterability. The effect of the primary characteristics on the secondary

characteristics was studied throughout the investigation. The iron particles for the different samples used were slightly porous and had a polycrystalline appearance. For powders which had rough particle surface, the apparent density was higher for the powder with higher average particle size, while for the powders which had relatively smooth particle surface, the apparent density was lower for the powder with high average particle size. The green density, densification parameter and sinterability of compacts made of fine powders were higher than those of compacts made of coarse powders.

**KEY WORDS:** iron powder, electrolysis, ferrous sulphate, particle size, particle shape, surface texture, apparent density, compressibility, sinterability.

## 1. INTRODUCTION

Particle technology is a highly complex subject, where fundamental scientific quantitative formulations and techniques are difficult to achieve [1, 2]. The main objective of powder metallurgy (P/M) is to produce final products with the highest possible uniform density. This, however, depends heavily on the initial loose powder characteristics [1, 3]. Characterization of powders help to control the powder quality during its manufacture or evaluate the powder to indicate its performance in (P/M) process [4-6]. The powder characterization parameters can be classified into two categories[1, 2, 7-10]: (i) the primary characterization parameters such as particle size, particle shape and particle structure (ii) the secondary characterization parameters such as apparent density, tap density, flow rate, interparticle friction, compaction and sintering characteristics. Some investigations [1, 2, 11-14] have been conducted to identify the significant primary characteristics of the powder which affect its secondary properties and to develop statistical methods and statistical probability density functions to explain the behavior of powders with irregular particles.

The present work aims to characterize and evaluate different samples of electrolytic iron powders prepared from the ferrous sulphate as a by-product of the iron and steel industry.

## 2. MATERIALS AND EXPERIMENTAL METHODS

### *2.1 Materials used*

Seven samples of electroytic iron powders were used in this investigation. These samples were prepared from ferrous sulphate by-product which is one of the wastes of the Egyptian Iron and Steel Company. The chemical analysis of ferrous sulphate used is given in Table 1. 0.3 M solution was prepared from this salt and the optimum conditions of the electrodeposition process in presence of different additives (to increase the cathodic current efficiency and consequently decrease the energy consumption) were determined elsewhere[15]. The deposited iron powder was thoroughly washed by distilled water and ethyl alcohol then dried in hydrogen atmosphere at 400° C for one hour. The produced powders will be denoted hereinafter as follows :

P (I) : The powder produced from a bath without additives.

P (II) : The powder produced from a bath containing 0.045g/l of carboxy methyl cellulose (C.M.C.).

P (III) : The powder produced from a bath containing 0.01 g/l of thiourea.

P (IV) : The powder produced from a bath containing 0.8g/l of ascorbic acid.

P (V) : The powder produced from a bath containing 0.5 g/l of urea.

P (VI) : The powder produced from a bath containing 0.5 g/l of glycine.

P (VII) : The powder produced from a bath containing 0.5 g/l of boric acid.

The iron content for all these powders was more than 99.4 %.

Table 1 Chemical analysis of ferrous sulphate salt

*2.2 Primary characterization parameters measurements*

Sieve size analysis and Scanning Electron Microscope (SEM) examination were used to characterize the particle size of the different iron powders.

In sieve analysis, a laboratory "Fritch" sieve shaker was used for dry screening of iron powders using a set of sieves of the following apertures: 1.5, 1.0, 0.7, 0.4, 0.25, 0.15, 0.1, 0.06 mm.

SEM of the type JEOL model JSM T20, Japan, was used for surface morphology inspection. The average particle size was calculated from the SEM photomicrographs as the diameter of the sphere of equivalent volume.

The particle shape for the different iron powders was examined using the SEM.

The particle surface texture for the different iron powders was examined by SEM, while the structure was examined by a reflected light metallurgical microscope.

*2.3 Secondary characterization parameters measurements*

The apparent density, the tap density and the flow time for the different iron powders were measured according to the standard procedures given in MNC handbook [16].

The different iron powders were moistened with 3% oleic acid as a lubricant, then equal weights of 4 g were pressed in a cylindrical mould of 10 mm inner diameter at 30 KN using a hydraulic press. The produced compacts were dried at 400°C for 15 minutes in hydrogen atmosphere. The compressibility is defined as the amount that a powder will compress or densify upon application of pressure (ASTM B 331). The densification parameter and compression ratio are used to define it [14].

$$\text{Densification parameter} = (\rho_{gr.} - \rho_{app.}) / (\rho_{th.} - \rho_{app.})$$

$$\text{Compression ratio at a specific pressure} = \rho_{gr.} / \rho_{app.}$$

Where,

$\rho_{gr.}$ is the green density.

$\rho_{app.}$ is the apparent density.

$\rho_{th.}$ is the theoretical density.

The green density of compacts was determined by dividing the weight of the green compact by its volume which was measured by displacement method.

Green compacts made of different iron powders were indurated in hydrogen at 900°C for one hour. The microstructures of these sintered compacts were studied using reflected light metallurgical microscope.

## 3. RESULTS AND DISCUSSION

Measurements of primary physical properties were performed in the different iron powders used. These measurements include particle shape, surface texture, size and structure.

Particle shape and size are two factors that are intimately connected. Without knowing the shape of a particle, it makes little sense to speak of particle size. The most common approach to describe and differentiate particle shape has been to use the dimensionality and surface contour of the particles [17].

SEM photographs of the different iron powders used are shown in Figs.1-7. It is obvious from Figs.1, 2 and 4 that each of the iron powders P (I), P (II) and P (IV) were in the form of agglomerates of irregular globular particles. The iron powder P (III) was in the form of a mixture of agglomerates of irregular globular grains and agglomerates of irregular and mostly elongated grains as shown in Fig.3. Figures 5-7 show that each of the iron powders P (V), P (VI) and P (VII) were in the form of agglomerates of laminated grains.

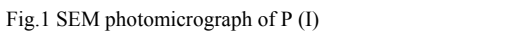

Fig.1 SEM photomicrograph of P (I)

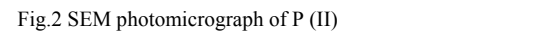

Fig.2 SEM photomicrograph of P (II)

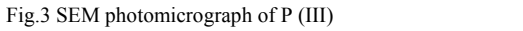

Fig.3 SEM photomicrograph of P (III)

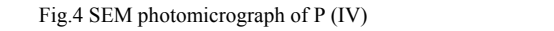

Fig.4 SEM photomicrograph of P (IV)



Fig.5 SEM Photomicrograph of P (V) 1000x



Fig.6 SEM Photomicrograph of P (VI) 500x

Fig.7 SEM Photomicrograph of P (VII) 1000x

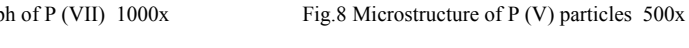

Fig.8 Microstructure of P (V) particles 500x

The laminated structure means that the crystals grow in preferred directions. The growth characteristics of crystals containing screw dislocations provide one explanation of preferred growth direction [18]. In case of the presence of additives to ferrous sulfate solution, the adsorption of additive molecules on preferred faces of the growing iron deposit, provides another explanation to the growth in the preferred orientations [19].

The SEM photomicrographs shown in Figs.1-7 indicate also that the surface texture of the iron particles of powders P (I), P (II) and P (III) was rough, while that of powders P (IV), P (V), P (VI) and P (VII) was relatively smooth.

Particle size and size distribution are fundamental properties, characterization of which is essential [20-22]. They affect the behavior of metal powders during processing; thus govern the properties of the final products made from powder. The particle size distribution determined by sieve analysis technique for the different iron powders is given in Table 2. From this table, it is obvious that both the powders P (I) and P (II) had a relatively wide particle size distribution range. The majority of their particles (more than 77% of total weight) had sizes lie in the range -1500 + 250 μm and the fractions - 1000 + 700 μm followed by - 400 + 250 μm were the highest percentages respectively. The powder P (III) had also a wide particle size distribution range, where about 63% had the size -250 + 60 μm and about 27% had the size - 1500 + 500 μm. The powders P (IV), P (V), P (VI) and P (VII) had a relatively narrow particle size distribution range, where more than 90% of each had a size of < 250 μm and the - 100 + 60 μm was the predominant fraction.

The average particle size for the different iron powders was calculated from data given in Table 2 (by standard statistical methods) and the results obtained are represented in Table 3. It is obvious from this table that the average particle size increased with addition of C.M.C. and considerably decreased in the presence of other additives.

Table 2 Sieve analysis of different iron powders

Table 3 Average particle size (A.P.S.) obtained using sieve analysis technique

The average particle size for different iron powders was also calculated from the SEM photomicrographs given in Figs.(1-7) and the results obtained are given in Table 4. It is obvious that

the average particle size obtained from SEM photomicrographs for different iron powders had the same trend of that obtained from the sieve analysis, but large differences are observed between them. These large differences are due to undisintegrated hard agglomerates in the sieve analysis samples.

Table 4 Average particle size (A.P.S.) obtained using SEM

Polished and etched sections of different iron powders particles were subjected to microscopic examination and photomicrographs of representative structures of some of them are given in Figs.8 and 9. This examination indicates that all types of iron powders particles were slightly porous and had a polycrystalline appearance.

*3.2 Secondary physical properties*

Apparent, tap densities, interparticle friction and flow time of the different iron powders are given in Table 5. It is obvious from this table that, for the powders P (I), P (II) and P (III) the apparent density was higher for the powder with higher average particle size. This is in good agreement with the results of Es-Saheb et al.[1]. On the other hand, for the powders P (IV), P (V), P (VI) and P (VII), the apparent density was lower for the powder with higher average particle size. This may be attributed to the smooth surface of the finer powders as shown in the SEM photomicrographs given in Figs.4-7. Powders with high apparent densities (such as P (II) and P (VI)) have the advantage that they allow shorter press-strokes and shallow die cavities thus reducing punch and die wear[14].

Fig.9 Microstructure of P (VI) particles 500x

Fig.10 Microstructure of sintered compact of P (I) 500x

Table 5 also shows that for the powders P (I), P (II) and P (III) the tap density had no definite trend with the average particle size due to the high roughness of their particles surface textures. On the other hand for the powder P (IV), P (V), P (VI) and P (VII), which had smooth particles, the tap density increased with the decrease in the average particle size. This result is in good agreement with the results of Es-Saheb et al.[1] and Uygur[14]. This is due to higher interparticle friction in the powder with small sizes as shown in Table 5. The ratio (tap density/apparent density) is an indicator of the interparticle friction. It is observed to be maximum for the powder P (VI) which had the finest particles.

The flow rate of powders becomes an important factor for rapid production of parts as the powder will be required to flow rapidly from storage containers to dies and also within the dies[14, 20, 21].

It is obvious from Table 5 that the flow time decreased with the decrease in the average particle size. This result seems to be strange, but this decrease in the flow time is due to the smooth surface of the finer powders as shown in SEM photomicrographs given in Figs.1-7.

Table 5 Apparent, tap densities, interparticle friction and flow time of different iron powders

The compressibility data for the different iron powders are presented in Table 6. This table shows that, both the green density and densification parameter of compacts made of powder P (IV), P (V), P (VI) and P (VII) were higher than those of compacts mode of powders P (I), P (II) and P (III). This is due to the higher percentage of fines in the powders from which the former compacts were made as shown in Table 2. These fines can enter and fill the voids between coarse grains during compaction. This is besides the high particle surface roughness of the powders from which the latter compacts were made as shown in SEM photomicrographs given in Figs.1-3. The high particle surface roughness makes the powder tends to absorb the lubricant added and consequently its effect in densification during compaction decreases.

Green compacts made of different iron powders were indurated in hydrogen at 900°C for one hour. The microstructures of some of the sintered compacts are shown in photomicrographs given in Figs.10-13.

Fig.11 Microstructure of sintered compact of P (III) 500x Fig.12 Microstructure of sintered compact of P (IV) 500x

For compacts made of the powder P (I) a semifused porous globular grains were observed and there was some evidence of direct bonding in the relatively large pores. For compacts made of the powders P (III), P (IV) and P (VI), the small pores were collapsed. The grain growth due to sintering can be easily observed. A comparative examination of the photomicrographs indicates that the number of grains per unit area decreased i.e. the average grain size increased with the decrease in the average particle size of the original powder. This is due to the increase in the green density and in turn the increase in the points of contacts between iron particles which consequently enhanced sintering and densification of compacts when subjected to induration at high temperature.

Fig.13 Microstructure of sintered compact of P (VI) 500x

Table 6 Compressibility data for different iron powders

## CONCLUSION

The average particle size increased with addition of C.M.C. and considerably decreased in the presence of other additives. The surface texture of the iron particles of powders P (I), P (II) and P (III) which are characterized by high average size was rough, while that of powders P (IV), P (V), P (VI) and P (VII) which are characterized by low particle size was relatively smooth. All types of iron powders particles were slightly porous and had a polycrystalline appearance. For powders P (I), P (II) and P (III) which had rough particle surfaces, the apparent density was higher for the powder with higher average particle size, while for the powders P (IV), P (V), P (VI) and P (VII) which had relatively smooth particle surfaces, the apparent density was lower for the powder with high average particle size. The flow time decreased with the decrease in the average particle size. The green density, densification parameter and sinterability of compact made of fine powders were higher than those of compacts made of coarse powders.

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