BIOBENEFICATION OF SILICA SAND FOR CRYSTAL GLASS INDUSTRY FROM ARDHUMA LOCATION, IRAQI WESTERN DESERT

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ABSTRACT

Silica sand from Ardhuma deposit in the Iraqi Western Desert has been biobeneficiated using mold *Aspergillus niger* to enhance its chemical properties, especially that associated with the removal of iron oxide impurities. The biobeneficiated silica sand obtained matches the required Fe_2O_3% level for crystal glass industry, which is less than 0.01%. The efficiency of Fe_2O_3 removal by bioleaching process; alone was 79.1%, the Fe_2O_3 content was 0.0125%. Whereas, combination of magnetic separation and bioleaching have improved the removal efficiency to 85.8%. The Fe_2O_3 obtained was 0.0085%. Silica sand produced by this approach have very low iron oxide content thus, it can be used for optical and crystal glass, as well as solar cell and semiconductors. The cost of bioleaching process was largely minimized as an alternative non-expensive source of carbon, which is Date extract used instead of highly expensive glucose.

استخدام الطرق البايولوجية لمعالجة رمال السليكا لإستخدامها في إنتاج زجاج الكرستال، منطقة أرضمة في الصحراء الغربية العراقية

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المستخلص

تم معاملة رمال السليكا لمنطقة أرضمة في الصحراء الغربية العراقية بالطرق البايولوجية باستخدام الفطر *Aspergillus niger* وذلك لتحسين المواصفات الكيميائية لهذه الرمال، خاصةً تلك المتعلقة بإزالة أوكسيد الحديد. ان رمال السليكا المحسنة توافق مستويات Fe_2O_3 المطلوبة لصناعة زجاج الكرستال والتي هي أقل من 0.01%، كما ان كفاءة إزالة أوكسيد الحديد بأستخدام طريقة الإذابة الحيوية (Bioleaching) لوحدها هي حوالي 79.1%، مع محتوى من أوكسيد الحديد حوالي 0.0125%. فيما أمكن تحسين كفاءة العملية بمزج عملية الإذابة الحيوية مع عدد من العمليات الفيزيائية، مثل الفصل المغناطيسي، حيث وصلت كفاءة العملية الى 85.8% وانخفض مستوى أوكسيد الحديد الى 0.0085%. كما تم في هذا البحث مضاعفة كميات الرمال المعالجة مقارنةً ببحث سابق وصولاً الى 100غم من الرمال بالطرق البايولوجية في الاختبار الواحد. وطبقاً للمستوى المنخفض المتحقق من أوكسيد الحديد في رمال السليكا باستخدام هذه الطريقة، فأن هذه الرمال يمكن استخدامها في صناعات عدة مثل صناعة الزجاج البصري وزجاج الكرستال، كما يمكن استخدامها في صناعة أشباه الموصلات وفي صناعة الخلايا الشمسية. بالإضافة الى ما تقدم، فقد أمكن في هذا البحث استخدام عصير التمر واطئ الكلفة بدلاً من سكر الكلوكوز باهض الكلفة في تنمية الفطر وبذلك أمكن اختزال كلفة العملية بشكل كبير.

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INTRODUCTION

In nature, noticeable microbial interaction with metals frequently manifests itself through metal immobilization or mobilization (Ferris *et al*., 1989; Ghiorse and Ehrlich 1992 and Ehrlich, 1996). Metal immobilization may be through cellular sequestration and accumulation, or through Extra-cellular precipitation. Metal mobilization results from dissolution of metal- containing phases. These processes are essential to control biological availability of metals in soils, sediment, and water (Ehrlich and Brierley, 1990).

Bioleaching of metals from ores is a practical example of the metal mobilization. The principle of bioleaching can be applied as biobenefication, a process in which an ore can be enriched in its valuable metal component(s) by selectively removing undesirable components. Biobenefication is a process whereby solid materials are refined and unwanted impurities are removed, using microorganisms and in this depends on their leaching activities. Some prokaryotes and eukaryotes may form metabolic products, such as acids or ligands that dissolve base metals of a certain minerals, such as Fe, Cu, Zn, Ni, Co and others. Others, may form anions, such as sulfides or carbonates that precipitate dissolved metal ions (Ehrlich, 1997).

Some bacteria and fungi can promote selective and non-selective leaching of one or more metals constituents from an ore or other rock with metabolic products, such as acids and/ or ligands. The acids may be organic or inorganic (Ehrlich, 1996).

Silica sand used for crystal glass production has certain permissible limits of impurities, especially iron oxide, which is considered the main limiting factor for the sand. However, the British Standards (BS 2975, 1988) classified silica sand used for glass production into 7 categories depending on the end uses; as shown in Table (1).

Table 1: British standards classification of sands by end-use

Grade	Type of sands	SiO_2 (%)	Fe_2O_3 (%)
A	Optical and Ophthalmic glass	99.7 Min	0.013 Max
B	Tableware and lead crystal glass	99.6 Min	0.010 Max
C	Borosilicate glass	99.6 Min	0.010 Max
D	Colorless container glass	98.8 Min	0.030 Max
E	Clear flat glass	99.0 Min	0.100 Max
F	Colored container glass	97.0 Min	0.250 Max
G	Glass for insulating fibers	94.5 Min	0.300 Max

PREVIOUS WORKS

Biological and non-biological treatments were investigated to purify silica sands from iron oxide impurities. Iraqi silica sands from Ardhuma deposit, which contain 0.09% Fe_2O_3 was purified by physical and chemical techniques including washing, sieving, magnetic separation and flotation, then treatment with (2 and 5)% HCl. The produced final sand has 0.016% Fe_2O_3 (Daykh and Mahdy, 2005).

Another approach; used a fixed-bed column leaching to treat Italian sand in an acid medium with a reducing agent (oxalic acid). This treatment decreased the Fe_2O_3 content from 0.03%, in the raw sands to 0.0163% (Ubaldini *et al*., 1996).

Biological methods were utilized for leaching iron from oxide minerals by means of microorganisms, therefore different heterotrophic bacteria and fungi are capable of dissolving iron from oxide minerals (Berthelin *et al*., 1974).

Bioleaching process combined with electromagnetic separation was applied to improve the quality of feldspar raw sand. As bacterial destruction of silicates was necessary to release the inter-granular spaces of silicate grains, the combination process decreased the iron content by 69% up to 74% (Iveta *et al*., 2005).

Combined bioleaching and electromagnetic separation, using *Bacills* spp. to decrease the iron content of Slovakian quartz sand by 60%. Thus, iron decreased from 0.135%, in the raw sand to 0.02% as Fe_2O_3 (Iveta *et al*., 2007)

Furthermore, another work had utilized bioleaching by the mold *Aspergillus niger* as well as a combination of multi process steps, starting from screening through magnetic separation and bioleaching as a final step. This method removes about 83.33% of the iron oxide content, meaning that the Fe_2O_3 content was decreased from 0.06% to 0.01%. While, the combination process, which constitutes of a preliminary treatment of the sands; followed by magnetic separation and bioleaching as a secondary treatment, removes 91.66% of the iron oxide content. The Fe_2O_3 concentration was decreased to 0.005% (Mustafa, 2008).

The aim of this study is to provide unconventional method to obtain highly pure silica sand for certain applications, like crystal glass production.

MATERIALS AND METHODS

▪ Materials

– The Sand Samples: Raw silica sands sample of 50 Kg from Ardhuma location was subjected to mixing, quartering and dividing to obtain representative samples of 1 Kg each. Consequently, one of these samples was screened on (850 and 150 μ) sieve opening to reject (+850 and −150 μ) sand fractions. Samples from the prepared sand (−850 +150 μ) were drawn for chemical and mineralogical analysis. The results are shown in Table (2).

Table 2: Chemical analysis of Ardhuma silica sand deposit

Chemical Composition (%)	**SiO_2**	**Fe_2O_3**	**Al_2O_3**	**CaO**	**TiO_2**	**SO_3**	**L.O.I**	**MgO**	**Na_2O**	**K_2O**
	97.9	0.06	0.47	<1	0.03	<0.07	0.43	0.06	0.02	0.01

– Biological Media and Chemicals: Yeast extract agar used as the semi-solid media for fungal cultivation. While the medium used for liquid cultivation contained alternative source of sugars that supply the mold with energy and carbon. This source was date extract, which was used instead of the highly expensive glucose. In addition, an optimization of Nitrogen source was conducted to insure maximum amounts of organic acids secretion. Nitrogen sources used are divided into organic compounds, such as urea and inorganic materials like ammonium nitrate and ammonium chloride. The media contained other components, but with minor doses; as shown in Table (3).

Table 3: Medium Components

Components	KCL	$MgSO_4$	KH_2PO_4
mg/l	25	250	500

Other materials being used in this study are:

-Growth medium: Yeast extracts broth.

-Hydrochloric acid (HCl), for adjusting pH of the culture solution before bioleaching step.

-Nutrient Medium: Potato, Dextiose, and Agar for insolent and activation of the mold.

-The mold used was *Aspergillus niger* isolated from wheat waste. The isolate was directed to multiple culturing to obtain pure isolates.

- **Methods**

– **Sieve Analysis:** One Kilogram of a representative sample was subjected to sieve analysis using sieve device type RETSCH and B.S sieve opening (850, 600, 250, 150, and 75)μ. The retained fractions were subjected to SiO_2 and Fe_2O_3 analysis to investigate the distribution of iron through the sand fractions. The results are shown in Table (4).

Table 4: Raw silica sand sieving analysis

Grain Size (μ)	Weight (%)	Accumulative Weight (%)		SiO_2	Fe_2O_3	Fe distribution
		Retain	Pass			
+ 850	0.12	0.12	99.88	–	–	–
–850+600	0.08	0.20	99.80	–	–	–
–600+250	32.60	32.80	67.20	99.20	0.03	16.300
–250+150	58.08	90.88	9.12	98.67	0.04	38.706
–150+75	8.96	99.84	0.16	96.05	0.24	35.840
–75	0.16	100	0.00	–	–	–

– **Magnetic Separation:** Samples of the size fraction (–600+150) μ and (–850+150) μ, of 100 gm each were subjected to magnetic separation, using dry high intensity magnetic separator type Outotech OUTO KUMPU Model MIH (13)111-5.

– **Biological Treatment:** The first stage of biological treatments consists of isolation and activation of the *Aspergillus niger* by using PDA medium. The activated mold, then inoculated to a solution medium, and incubated until fixed pH was obtained. Several factors have been determined for the mold growth rate, biomass production and secretion of organic acid (mainly citric and oxalic acids) through the monitoring of daily pH recordings. After inoculation these factors include: Carbon source of the solution media, presence and absence of yeast extract in the mold media, starting pH prior to incubation (7.0 and 5.5) and incubation temperature (Room and 20)º C.

The second stage included the bioleaching process, which is in general accomplished by adding 100 gm of silica sand sample to 250 ml conical flask containing 100 ml of the mold (biomass free) solution. Then was mixed by electrical mixer for different periods of time and temperatures. Then the treated sand was washed and filtered thoroughly and finally dried in oven at 100° C. The biobeneficiated sand was subjected to chemical analyses, using atomic absorption to determine the removal efficiency of Fe_2O_3. The main parameters that affect on the bioleaching process include grain size range [(–850+150) μ and (–600+150) μ], temperatures of bioleaching (25 and 100)° C, time periods (2, 3 and 4) hr, pH adjustment, the addition of 10% HCl to the sand, before bioleaching and the source of the nitrogen.

– **Combined Treatment:** Pre-processed samples were subjected to biological processing using growth culture solutions of the mold *Aspergillus niger*, these treatments occasionally were combined with acid leaching of 10% HCl or pH adjustment to 0.5, prior to bioleaching.

RESULTS AND DISCUSSION

- **Pre-Bioleaching parameters**

Several parameters were assayed to evaluate their effects on the removal of iron impurities from the silica sands. The values of pH were used as indicators for evaluation of the growth media.

– **Effect of Source Carbon:** Several media were prepared using three types of carbon source (glucose, sucrose and date extract). These media were monitored after 7 days of inoculation and evaluated according to the pH values obtained. The results shown in Table (5) indicate that the media contain date extract as a carbon source had the lowest pH value after a week of incubation, which is conflicting high secretion of organic acids mainly (citric and oxalic). In addition, the used date extract as a carbon source enhanced the mycelium growth produced with a larger biomass production, which is highly maturated in its reproductive stage (spore formation). According to the results shown in Table (5), the date extract seems to be the best source of carbon that can be used for the mold growth, due to low achieve pH value (high organic acids secretion). This might be resulted from the wide types of nutrients content in date extract. In addition, the date extract is considered to be of low cost in comparison with glucose and sucrose.

Table 5: Effect of carbon source on the final pH of the culture

Media of carbon source	pH after 7 days of incubation
Glucose	1.90
Sucrose	2.05
Date extract	1.70

– **Effect of Yeast Extract:** Yeast extract was used as a promoter for the growth of fungi (molds). It was noticed that mold's biomass has a negative influences in the presence of yeast extract; when date extract was used as a source of carbon for the media, contrary to that of glucose and sucrose. The removal of iron oxide showed similar results in the presence and absence of yeast extract as it is shown in Table (6). Thus, yeast extracts were excluded from the next experiments to reduce the cost of treatment.

– **Effect of Initial pH:** Two starting pH values were used (7.0 and 5.5). The results shown in Table (6) indicate an increase in the removal of iron oxide, with the increasing of pH from 5.5 to 7.0. Thus, in the next experiments starting pH of pH 7 was used.

– **Effect of Incubation Temperature:** To evaluate the role of incubation temperature on the organic acids production from the mold isolated *Aspergillus niger*, the mold was cultured into two incubation temperatures (room and 20° C). The results in Table (6) reveal that, in most cases, the considered room temperature being quite enough to provide suitable condition for organic acids production. This will eliminate the need for temperature controlling during incubation.

Table 6: Effect of the yeast extract on the final pH

Yeast extract	Incubation temperature	Starting pH	Final pH
Presence	20º C	5.5	1.90
		7.0	1.87
	Room Temperature	5.5	1.82
		7.0	1.80
Absence	20° C	5.5	1.87
		7.0	1.77
	Room Temperature	5.5	1.75
		7.0	1.65

▪ **Bioleaching Parameters**

Four parameters were believed to be played a vital role in the bioleaching step; some of these parameters represent a combination of bioleaching with other processes, like addition of hydrochloric acid, pH adjustment and magnetic separation prior to bioleaching.

– **Effect of Temperature of Bioleaching and Sand Grain Size:** To study the effect of sand particle size on the bioleaching process on iron oxide removal, two size fractions were prepared for bioleaching at two different temperatures (25 and 100° C). The results illustrated in Fig. (1) show that, the higher removal of iron oxide was achieved using sand with particle size ranges of (–600 +150) µ, then with sand of particle size ranges (–850 +150) µ. Therefore, (–600 +150) µ was subjected for the next experiment.

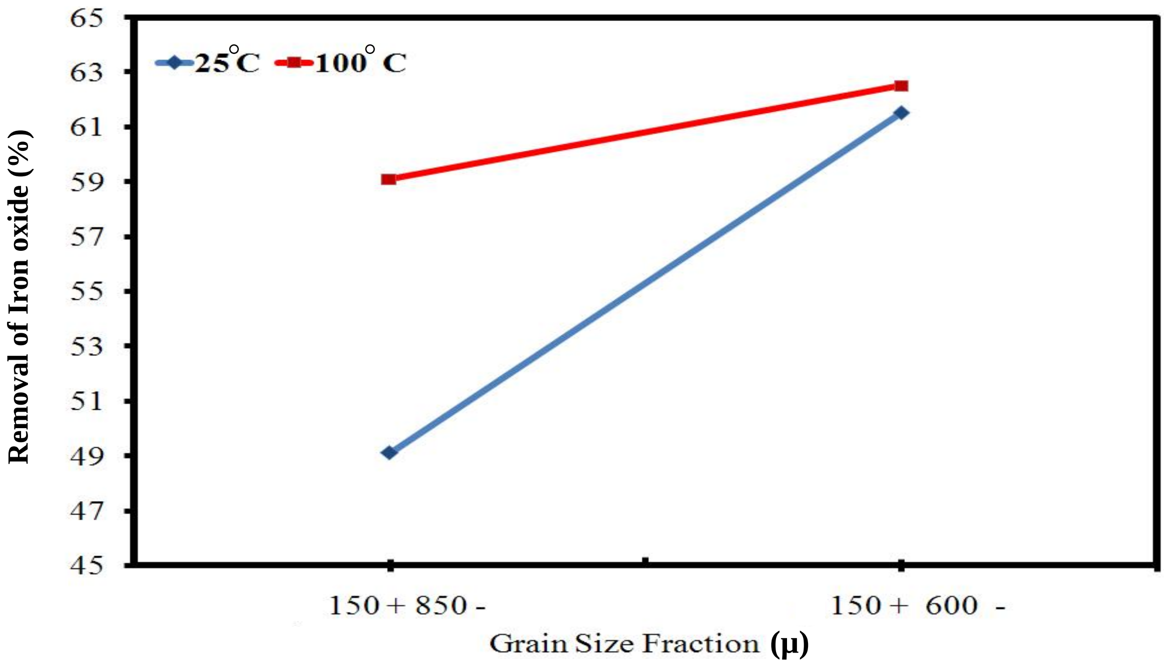

Fig.1: Effect of sand grain size on the Fe_2O_3 removal (25° and 100° C)

– **Effect of pH Adjustment:** It had been previously demonstrated that bioleaching can be enhanced through pH-adjustment (Mustafa, 2008). In this study, the pH was adjusted using concentrated HCl to treat the silica sand fraction (–600 +150 µ). The results are shown in Fig. (2) that reveal an iron oxide removal, which is largely increased as the pH was adjusted to 0.5. This case occurred only at boiling temperature, while there was very low effect of pH-adjustment at room temperature.

– **Effect of Combination of Bioleaching with Magnetic Separation:** The silica sand for optical glass production requires Fe_2O_3 content less than 0.013%, and less than 0.010% for crystal glass, and semiconductors grade. Treatment of the sand by bioleaching alone removes about 79% of iron oxide, decreasing Fe_2O_3 contents from 0.06%, in the raw sand to 0.012%, which is not acceptable for crystal glass product. Thus, bioleaching needs to be combined with another suitable process; such as magnetic separation to obtain a product with Fe_2O_3 less than 0.01%. The achieved removal of iron oxide was of about 83.50%.

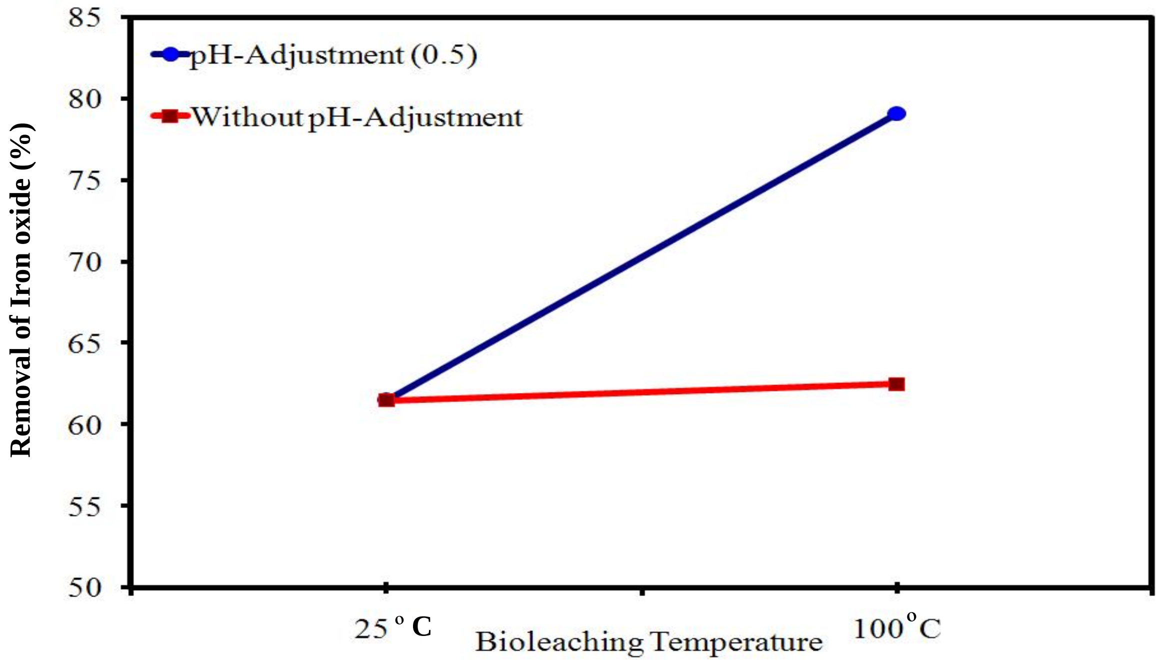

Fig.2: Effect of pH-adjustment on bioleaching

Magnetic separation was carried out with magnetic field intensity of about 16.5 Kilogauss, to improve the removal of Fe_2O_3. The non-magnetic portion of the sand was used for series of experiments including times bioleaching at (2, 3 and 4 hours). The experiments were conducted at sand fraction (–600 +150) µ under boiling temperature and the pH was adjusted to 0.5. The results are shown in Fig. (3), it is obvious that 4 hours of bioleaching using the above conditions gave removal of iron oxide of 85.16%, lowering the Fe_2O_3 content to about 0.0089%. Whereas, 3 hours of bioleaching, resulted silica sand with Fe_2O_3 content of 0.0098% with a removal of about 83.66% iron oxide. At 2 hours of bioleaching, the achieved removal of iron oxide was of 83.16%, lowering of the Fe_2O_3 content to 0.0101%, which is a little bit more than the maximum limits required for crystal glass production. From the aforementioned results, we can conclude that removal of iron oxide increase directly with the increasing of bioleaching duration.

Another set of experiments conducted under the same conditions and variables ,except the pH-adjustment step, which was replaced by the addition of 50 ml of 5% HCl, and then bioleaching proceeds for 2, 3 and 4 hours. The results in Fig. (3) show that the addition of 5% HCl with 4 hours of bioleaching improved the removal of iron oxide up to 85.83% and resulted in decrease of the Fe_2O_3 content to 0.0085%. Since 3 hours of bioleaching produced sand with Fe_2O_3 content acceptable for crystal-grade sand, and due to economical consideration, it can be considered more suitable than that of 4 hours of bioleaching.

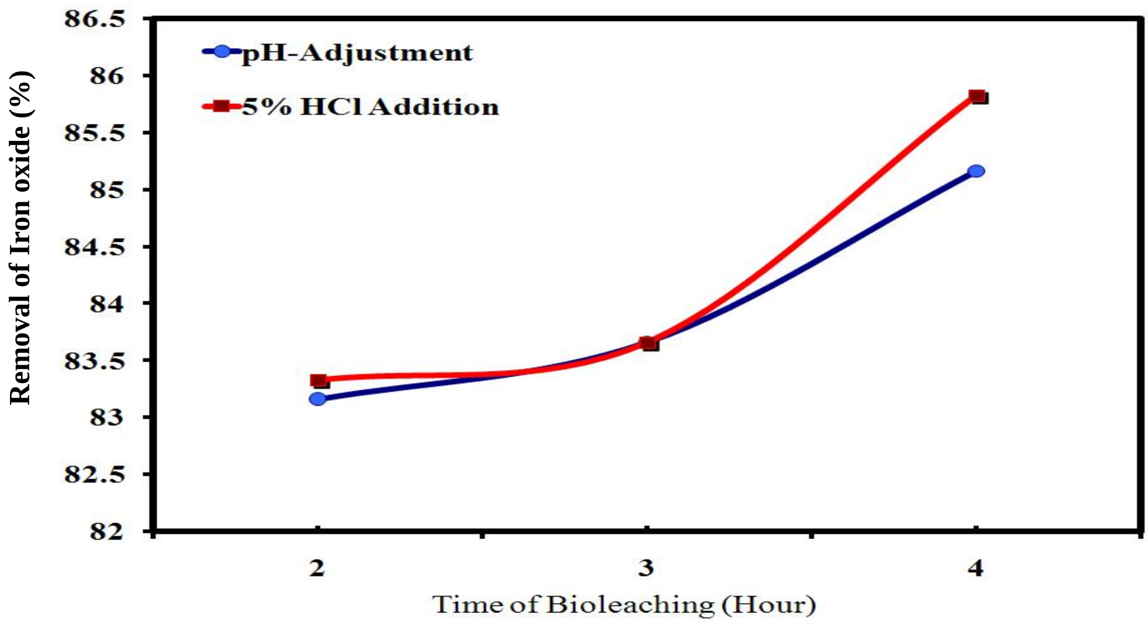

Fig.3: Effect of bioleaching time on the combined process

- **Effect of Nitrogen Source for the Media:** Among the essential nutrients for the growth of molds are nitrogen compounds, and its believed that molds secretion of organic acid is deeply influenced by the type of the used nitrogen source (Al-Hamando, 1989). Hence, nitrogen source could be organic, such as urea or inorganic like ammonium nitrate and ammonium chloride. Therefore, these compounds were tested to determine their effect on the organic acid secretion (mainly citric and oxalic) by *Aspergillus niger*, this was done by recording the final achieved pH value, which intern is directly proportional with Fe_2O_3 removal (Table 7). These results obviously pointed out that ammonium nitrate, which is used almost in all bioleaching experiments was the best nitrogen source for organic acids secretion and Fe_2O_3 removal. Thus efficiency of bioleaching was increased according to the following path Ammonium Nitrate > Urea > Mixture > Ammonium Chloride. This might be due to the effect of each nitrogen source on the organic acid synthesis pathways.

Table 7: Effect of nitrogen source on the Fe_2O_3 removal

Nitrogen Source	Final pH	Fe_2O_3 Removal (%)
Ammonium Nitrate	1.65	85.16
Ammonium Chloride	1.74	81.00
Urea	1.70	84.00
Mixture	1.72	82.50

Some of the final products of combination processes were subjected to complete chemical analysis; the results are shown in Table (8). The results indicate that silica sand of crystal grade can be obtained by combining magnetic separation and microbial activities, especially those associated with secretion of several organic acids that can leach iron oxide and prevent it from re-adsorption on the sand particles again. The results also outlined the possibility to modify and improve the process of bioleaching by addition of mineral acid as well as pH-adjustment.

Table 8: Chemical composition of selective products of the combined treatments

Products	Chemical analysis (%)										
	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	CaO	MgO	SO_3	L.O.I	Na_2O	K_2O	Cr_2O_3
BS (1)	99.62	0.0089	0.07	0.03	0.10	0.016	< 0.07	0.27	0.03	0.01	N.D
BS (2)	99.70	0.0085	0.10	0.02	0.10	0.014	< 0.07	0.16	0.02	0.01	N.D
BS (3)	99.71	0.0096	0.11	0.02	0.10	0.016	< 0.07	0.11	0.04	0.01	N.D
BS (4)	99.80	0.0096	0.10	0.02	0.10	0.015	< 0.07	0.08	0.00	0.00	N.D

Where

BS (1): non-magnetic portion, pH-adjusted (0.5), 100º C, 3 hours and Ammonium Nitrate as nitrogen source
BS (2): non-magnetic portion, 50 ml of 5% HCl, 100º C, 3 hours and Ammonium Nitrate as nitrogen source
BS (3): non-magnetic portion, 100º C, 3 hours, and Urea as nitrogen source
BS (4): non-magnetic portion, pH-adjusted (0.5), 100º C, 3 hours and Urea as nitrogen source

CONCLUSIONS

- Silica sands for crystal and semiconductors industries, as well as solar glass grade can be produced using bioleaching process combined with high intensity dry magnetic separation.
- Date extract, as a cheap source for carbon, when used as an alternative source for the highly expensive Glucose or Sucrose revealed better results for fungus growth, final pH and Fe_2O_3 removal.
- The optimum conditions for crystal-grade silica sand could be achieved by treating the sand fraction of (−600 + 150) μ, with magnetic separation using magnetic intensity of 16.5 Kilogauss, then pH-adjustment or addition of 5% HCl to the growth solution, and finally bioleaching at 100º C for 3 hours.
- Carbon and nitrogen source of the media played a vital role in the secretion of organic acids that are responsible about leaching out the iron oxide from the silica sand, thus date extract and ammonium nitrate or urea are the best sources of carbon and nitrogen, respectively.

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