

REMOVAL OF SOME POLLUTING METALS FROM INDUSTRIAL WATER USING CHICKEN FEATHERS

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ABSTRACT

This work aimed to use of chicken feathers (CF), as a natural source of active amino acids after alkaline treatment with 0.95N NaOH or 0.6N NaOCl solution and immobilization on silica surfaces as regenerable sorbents, for the removal of polluting calcium, magnesium, iron and manganese from industrial water sectors. Batch sorption and sorption isotherms were performed to evaluate the effect of sorption process parameters, such as pH, time, sorbent amount, metal concentration, temperature, interfering anions and metal selectivities. The results obtained revealed that, 1.0g of NaOH-treated CF-immobilized silica remove 93.38% Ca, 72.55% Mg, 97.13% Fe and 95.66% Mn, while, 1.0g of NaOCl-treated CF-immobilized silica remove 92.14% Ca, 71.85% Mg, 94.26% Fe and 93.57% Mn at pHs 9, 8.5, 6 and 7, within 20-30 minutes, respectively. Generally, the NaCl regenerable sorbents were applied successfully for treating industrial water samples in different Egyptian industrial areas.

1. INTRODUCTION

During the past decades, many works had been done to produce a novel composition of matter to remove polluting metals such as, calcium, magnesium, iron and manganese from industrial water systems such as, boilers, heat exchangers, chemical reactors, cooling towers and reverse osmosis (RO) plants. These polluting metals lead to serious economic industrial hazards fall into: precipitation of scale, deposition of suspended matters, corrosion, system efficiency loss, increased energy

consumption and emergency shutdown [Hasson et al., (1968); Jackson et al., (1973); Shams et al., (1989) and Al-Ahmad et al., (1993)].

Commercially available ion exchange resins are currently used in water treatment to produce soft water. However, some resins suffer from disadvantages of being expensive and/ or being difficult to produce [Vassiliou et al., (1968) and Varmeulen et al., (1983)].

Recently, many researches in the environmental science are concerned in the production of inexpensive regenerable sorbents of high exchange capacities from different natural wastes in either native or chemically modified form [Brezny et al., (1988); Orham et al., (1993); Metwally, (1994); Spinti, (1995); Horsfall et al., (2003) and Palma et al., (2003)]. In literature, some inventions used proteinaceous materials selected from the group consisting of hair, hoof, horn, wool and feathers to remove some pollutant metals [Tan et al., (1985); Kawaguchi, (1996); Homonoff, (2002) and Al-Ashch et al., (2003)].

Poultry chicken feathers represent about 6.0% of the total weight of mature chicken lead to environmental problems as waste – by product at commercially poultry plants [Dalev, (1994); Dyke et al., (2001); Allen, (2002) and Stoltz, (2003)]. CF are bio-source with high keratinaceous protein content (more than 750 g kg⁻¹ crude protein). So, after alkaline treatment of CF with NaOH or NaOCl solution provides active amino acids which on immobilization on silica activates its surface towards metal uptake.

2. MATERIALS AND METHODS

All chemicals used for preparation of metal solutions were of analytical grade viz. CaCl₂, MgCl₂.6H₂O, FeCl₂.4H₂O and MnCl₂.4H₂O (Sigma-Aldrich Co.). All metal solutions were prepared using doubly distilled water (18.5 MΩ.cm⁻¹). NaNO₃, Na₂SO₄, NaHCO₃ and NaCl were of analytical grade (Koch-Light Co.). NaOCl (14%), petroleum ether (40-60°C), ethanol (95%) and Silica (60 mesh) (El-Nasr Co.).

2.1. Sorbents preparation

2.1.1. Cleaning and preparation of CF

CF were collected from poultry slaughterhouses and cut into small pieces. The dirt and impurities attached to CF were removed using petroleum ether (40-60°C) for 20 min. filtered off, washed with tap water

and ethanol (95%) for several times, and left to dry overnight to get rid of any unpleasant odor.

2.1.2. Treatment with alkaline solutions [NaOH or NaOCl]

2.0g of CF were stirred at 80 rpm with 20 ml 0.95N NaOH at 70°C for one hour, or 0.6N NaOCl at 80°C for two hours. The produced solutions were filtered to remove any ash and wax residues. Analysis of the resultant amino acid hydrolyzates was carried out using amino acid analyzer (LC 3000-Eppdraf-Germany).

2.2. Immobilization of amino acid residues on silica

The produced hydrolyzates having high pH should be lowered to about 2 with 1N HNO₃. 2.0g Silica (60 mesh) was shaken at 50 rpm with either NaOH or NaOCl amino acids hydrolyzates at 60°C for two hours and used as sorbents in this study [Malachowski et al., (2004)].

2.3. Batch sorption experiments

1.0g sorbent was shaken with 10 ml of 6.75% NaCl solution at 150 rpm, at room temperature for 20 min, then rinsed with doubly distilled water several times to remove any metal traces attached to active amino acid sites on sorbent. The metals under investigation were determined using Plasma Optical Emission-Mass Spectrometer [(OPEMS III)-Thermo Elemental Co.] after shaking their solutions with sorbent (1% wt/v.) at 150 rpm, within various times at optimum pH for each metal.

2.4. Equilibrium isotherm experiments

Equilibrium isotherm was performed by shaking metal solutions of different concentrations (50 to 1000 mg/l) with sorbent (1% wt/v.) at 150 rpm, at room temperature within the optimum time and pH for each metal ion to ensure the attainment of equilibrium.

3. RESULTS AND DISCUSSION

3.1. Alkaline treatment conditions

CF amount, NaOH or NaOCl concentrations, temperature and time for CF hydrolysis yielding maximum total amino acid concentrations were examined. Table 1 shows that, total amino acid concentrations of hydrolyzate were increased as NaOH or NaOCl concentration increase till it reaches 0.95N or 0.6N respectively. This

may be due to decrease in cross-linkage of CF keratin and hydrolysis of peptide bonds yielding active amino acid residues. It is also observed that, as CF amount, temperature and time increased till reach (2.0g, 70°C, 1h for NaOH treatment and 2.0g, 80°C, 2h for NaOCl), total amino acid concentrations increased, due to the enhancement in keratin hydrolysis rate. However after these values, some decrease in total amino acids concentration was observed, which may be due to the destruction and racemization of some amino acids as threonine, arginine and cysteine in highly alkaline environment [Hugli, (1972) and Fountoulakis et al., (1998)].

Table (1): Effect of CF amount, NaOH or NaOCl concentration, temperature and time on CF alkaline treatment.

	NaOH treatment					NaOCl treatment				
a) Alkali conc., mole/l	0.5	0.75	0.85	0.95	1.0	0.4	0.5	0.55	0.6	0.7
Total amino acids conc., mg/l	258.6	281.2	290.7	298.9	296.5	249.8	263.1	275.9	287.3	281.4
b) Temperature, °C	50	60	70	80	90	50	60	70	80	90
Total amino acids conc., mg/l	298.8	321.2	334.0	333.1	331.5	287.3	292.0	309.8	316.4	314.7
c) Time, min.	30	60	90	120	150	30	60	90	120	150
Total amino acids conc., mg/l	293.4	334.0	333.9	333.2	333.4	280.5	316.4	321.3	328.7	327.9
d) CF amount, g	1.0	1.75	2.0	2.25	2.5	1.0	1.75	2.0	2.25	2.5
Total amino acids conc., mg/l	169.7	298.2	334.0	334.1	334.0	161.3	290.5	328.7	328.6	328.6

a) Effect of Alkali concentration, using 2.0g CF, at 50°C, for 1h.

b) Effect of Temperature, using 2.0g CF, 0.95N NaOH or 0.6N NaOCl for 1h.

c) Effect of time, using 2.0g CF, 0.95N NaOH at 70°C or 0.67N NaOCl at 0°C.

d) Effect of CF amount, using 0.95N NaOH at 70°C for 1h or 0.6N NaOCl at 80°C for 2h.

The obtained results (Fig.2 and 3) were confirmed by comparing with amino acid analysis after hydrolysis of 2.0mg CF with 4ml 6N HCl at 110°C for 24h (Fig.1) according to [AOAC, (1984)]. No great differences were observed.

3.2. Metal-sorption capacity

3.2.1. pH effect

The solution pH was shown to be the key parameter influencing the structural formula of active amino acids molecules bonded to silica and consequently the coordination properties of the prepared sorbents. The general structural formula of most natural amino acids contain both (-COOH) and (-NH₂) groups, which give them the ability to exist either as a cation, anion, or zwitterion depending on pH. For this reason pH governs the ionic form of the active amino acid sites occurred on silica.

The efficiency of the sorption of Ca, Mg, Fe and Mn on prepared sorbents was examined at pH from 1 to 9 (Fig.4). It was observed that, the maximum uptake of Ca, Mg, Fe and Mn were at pH 9, 8.5, 6 and 7, respectively. From this we can assume that, in pH more than isoelectric point (isoelectric point is pH at which amino acid becomes neutral), anion form of amino acids occurred on silica binds Ca and Mg through the amino nitrogen atom and the carboxyl oxygen atom forming stable chelating ring [Schmidbauer et al., (1989)].



While in case of Fe and Mn sorption, the metal is coordinated through carboxyl oxygen atom of amino acid sites but is not coordinated through the nitrogen atom [Inomata et al., (1984) and Farkas et al., (1990, 1993)]. Metal can be also coordinated to side chain charged groups (-OH, -COOH and -SH) of some amino acid depending on type of amino acid to complete its coordination sphere [Osamu et al., (2002)].

3.2.2. Time effect

As shown in (Fig.5), it was observed that, sorption equilibrium of Ca, Mg, Fe and Mn on NaOH-treated CF-immobilized silica was attained after 20, 20, 20 and 25 minutes, respectively; while their sorption equilibrium on NaOCl-treated CF-immobilized silica was attained after 25, 25, 30 and 30 minutes.

3.2.3. Sorbent amount effect

As shown in (Fig.6), it was observed that, the sorption of metal ion is dependent on the amount of sorbent used. The uptake of Ca, Mg, Fe and Mn were gradually increased with sorbent amount (0.2 to 1.5g)

till most of metal ions in solution were be completely removed in the range (1-1.5g) of sorbent.

3.2.4. Temperature effect

(Fig.7) Shows that, the increase in temperature of metal solution enhances the sorption of Ca, Mg, Fe and Mn on sorbents. This may be due to an enhancement of the endothermic dehydration of water spheres around metal ions and consequently facilitates metal sorption on sorbents. After optimum temperature of sorption equilibrium, the metal sorption is clearly decreased because the mobility of metal increased leading to a decrease in the feasibility of metal ion to interact with active amino acid sites on sorbent.

3.2.5. Sorption isotherm

The results of equilibrium isotherms for sorption of Ca, Mg, Fe and Mn on prepared sorbent are shown in (Fig.8). The results are described well by Freundlich isotherm model, whereas the initial concentration of metal solution increased, the sorption capacity of sorbents increased, and no region of discontinuity was observed.

$$\ln Q_e = \ln K_f + 1/n \ln C_e$$

where,

Q_e (mmole/g) : metal uptake at equilibrium.

C_e (mmole) : metal concentration of the equilibrium solution.

$1/n, K_f$: represent the slope and intercept of the linear plot, and related to sorption intensity and sorption capacity respectively.

From linear plots, it is found that there were no single points indicating that, the sorption process is completed. The Freundlich constants for both sorbents are presented in Table 2. From the values of K_f , it can be concluded that NaOH-treated CF-immobilized silica have greater sorption capacity than NaOCl-treated CF-immobilized silica.

Table (2): The Freundlich constants for metal sorption on sorbents.

Metal	NaOH-treated CF-immobilized silica			NaOCl-treated CF-immobilized silica		
	K_f	1/n	R^2	K_f	1/n	R^2
Ca	0.3616	0.5236	0.9834	0.3446	0.5462	0.9890
Mg	0.3944	0.5079	0.9845	0.3867	0.5381	0.9871
Fe	0.2432	0.5764	0.9924	0.2330	0.5948	0.9956
Mn	0.2410	0.5656	0.9947	0.2358	0.5875	0.9962

3.2.6. Anions interference

Changes in the uptake of Ca, Mg, Fe and Mn by sorbents in the presence of different concentrations of nitrate (0.5-2.5 mg/l), bicarbonate (130-330 mg/l) and sulfate (30-2000 mg/l) were being investigated in Table 3. It was observed that, an increase in the concentration of nitrate, bicarbonate and sulfate leads to a very little increase in the metal uptake. The reasonable assumption is that, the anions carrying negative charges accept protons, which make competition for ion binding of metal by sorbent sites. However, after certain concentrations of sulfate which differ from nitrate and bicarbonate by its higher negative charges, the metal uptake fall down due to dissolution of some active amino acids bonded to silica.

Table (3): The Effect of anions on the metal uptake efficiency on sorbents.

Interfering anion	NaOH-treated CF-immobilized silica				NaOCl-treated CF-immobilized silica			
	Ca uptake %	Mg uptake %	Fe uptake %	Mn uptake %	Ca uptake %	Mg uptake %	Fe uptake %	Mn uptake %
Bicarbonate conc., mg/l								
130	93.72	72.83	97.25	95.83	92.56	71.23	93.64	93.62
180	94.15	73.47	97.41	92.95	92.85	71.58	93.23	94.07
230	94.89	73.61	98.09	96.67	93.17	72.74	94.02	94.91
280	95.26	74.26	98.15	97.55	93.52	72.94	94.95	95.64
330	95.95	75.14	98.73	98.38	94.16	74.87	96.13	96.98
Nitrate conc., mg/l								
0.5	93.41	72.53	97.15	95.47	92.25	71.94	94.08	93.52
1.0	93.59	72.66	97.37	95.64	92.84	72.26	94.55	93.68
1.5	94.96	73.12	97.45	96.93	93.57	72.46	94.71	94.24
2.0	95.60	73.76	97.81	98.25	94.43	73.51	95.98	94.57
2.5	96.12	74.90	98.46	98.47	95.27	73.97	96.42	95.11
Sulfate conc., mg/l								
50	93.05	72.06	96.95	95.45	91.94	70.13	94.83	93.62
500	96.81	75.29	98.57	97.08	92.17	71.68	95.25	94.93
1000	90.14	70.58	98.82	93.59	87.11	67.12	90.71	96.75
1500	86.79	64.56	90.93	86.75	82.35	59.94	85.08	91.06
2000	84.13	60.03	81.74	80.3	79.72	54.78	79.86	83.64

3.2.7. Selectivities

Selectivity of each metal ion, at its optimum sorption pH value on sorbents was being examined. Table 4 shows that pH plays an important role in investigation of the metal sorption selectivity. It was observed that, as pH rise to 8.5 and 9, the sorbents become more selective for Mg and Ca and less selective for Fe and Mn. However, Ca and Mg suffered from some interferences at their optimum pH due to similarity of them in their coordination behaviors with sorbents and hydroxide precipitation of Fe and Mn blocking sorbent surface. As pH lowered to 7 and 6 the sorbent become more selective for Mn and Fe and less selective for Ca and Mg. However, Fe and Mn suffered from interferences.

Table (4): Metal ion sorption selectivity on sorbents as a function of solution pH.

pH	NaOH-treated CF-immobilized silica				NaOCl-treated CF-immobilized silica			
	Ca uptake %	Mg uptake %	Fe uptake %	Mn uptake %	Ca uptake %	Mg uptake %	Fe uptake %	Mn uptake %
9	88.87	24.61	99.93 ^a	99.48 ^b	84.21	20.72	99.95 ^a	99.61 ^b
8.5	32.19	67.43	99.16 ^a	98.92 ^b	29.74	66.58	99.4 ^a	99.27 ^b
7	13.06	6.27	16.91	85.67	11.24	4.68	12.7	82.75
6	10.75	5.12	89.24	14.98	8.06	2.95	86.03	16.92

a. Iron precipitation.

b. Manganese precipitation.

3.2.8. Sorbent recovery (regeneration)

Regeneration process of the prepared sorbents is very important economic process in which the metal ions already bonded to sorbent were stripped (liberated) for reuse sorbent several times. Ten ml of NaCl solutions of different concentrations from 2.5% to 7.0% were shaken at 150 rpm with 1.0g sorbent at room temperature for 20 minutes till all metal ions stripped from sorbent. As shown in (Fig. 9), maximum concentration of NaCl solution required to strip Ca, Mg, Fe and Mn from NaOH-treated CF-immobilized silica were 5.75%, 5.5%, 6.5% and 6.0% respectively, while maximum concentration of NaCl solution required for NaOCl-treated CF-immobilized silica were 5.5%, 5.5%, 6.0% and 5.75% respectively. NaCl solution is preferred as regenerating solution, due to its availability and low cost.

3.3. Sorbent application

Both regenerable sorbents were be applied successfully to remove most of Ca, Mg, Fe and Mn from industrial water samples of different Egyptian factories. Table 5 shows that, 98.1% and 96.5% of 77.1 mg l⁻¹ Fe in El-Zamel Steel manufacture wastewater (industrial zone of 6th October City), 96.8% and 94.9% of 83.6 mg l⁻¹ Fe in El-Attaal steel manufacture wastewater (industrial zone of Suez City) were removed at pH 6.0 using 1.0g of NaOH-treated CF-immob. silica, or NaOCl-treated CF-immob. silica, respectively. Both factories suffer from economic and environmental effects from high concentration of toxic iron. 95.6% and 92.1% of 97.3 mg l⁻¹ Ca in AC&T Chemical manufacture wastewater (industrial zone of 6th October City), 54.1% and 52.5% of 172.8 mg l⁻¹ Ca

in brackish feed water of DELTA RO. Plant (El-Sadat City) were removed in the pH range 8.5-9.0 using 1.0g of NaOH-treated CF-immob. silica or NaOCl-treated CF-immob. silica, respectively. Both plants suffer from scale and corrosion problems.

Table (5): Application of sorbents in the removal of polluting Ca, Mg, Fe and Mn from industrial water.

Parameters of analysis	pH	E.C mg/l	Ca mg/l	Mg mg/l	Fe mg/l	Mn mg/l	HCO ₃ ⁻ mg/l	SO ₄ ²⁻ mg/l	Cl ⁻ mg/l	NO ₃ ⁻ mg/l
El-Zamel Steel manufacture wastewater										
Water sample source	7.6	234	34.6	11.2	77.1	2.65	162	43.1	38.2	1.1
NaOH-treated CF-immob. Silica (metal uptake %)	6.0		76.4%	89.3%	98.1%	96.0%				
NaOCl-treated CF-immob. silica (metal uptake %)	6.0		75.1%	86.6%	96.5%	94.9%				
El-Attaal steel manufacture wastewater										
Water sample source	7.3	241	39.5	16.3	83.6	4.91	145	39.4	40.6	2.1
NaOH-treated CF-immob. silica (metal uptake %)	6.0		74.8%	82.7%	96.8%	92.1%				
NaOCl-treated CF-immob. silica (metal uptake %)	6.0		71.3%	78.9%	94.9%	90.8%				
AC&T Chemical manufacture wastewater										
Water sample source	8.1	259	97.3	36.4	0.7	0.45	182.9	58	39.3	1.7
NaOH-treated CF-immob. silica (metal uptake %)	9		95.6%	91.7%	99.5%	98.8%				
NaOCl-treated CF-immob. silica (metal uptake %)	9		92.1%	89.8%	99.1%	97.3%				
DELTA RO. Plant Brackish feed water										
Water sample source	8.5	2464	172.8	67.1	8.3	5.9	327	521	1481	2.4
NaOH-treated CF-immob. Silica (metal uptake %)	8.5		54.1%	70.6%	99.8%	99.1%				
NaOCl-treated CF-immob. silica (metal uptake %)	8.5		52.5%	66.9%	99.3%	98.5%				

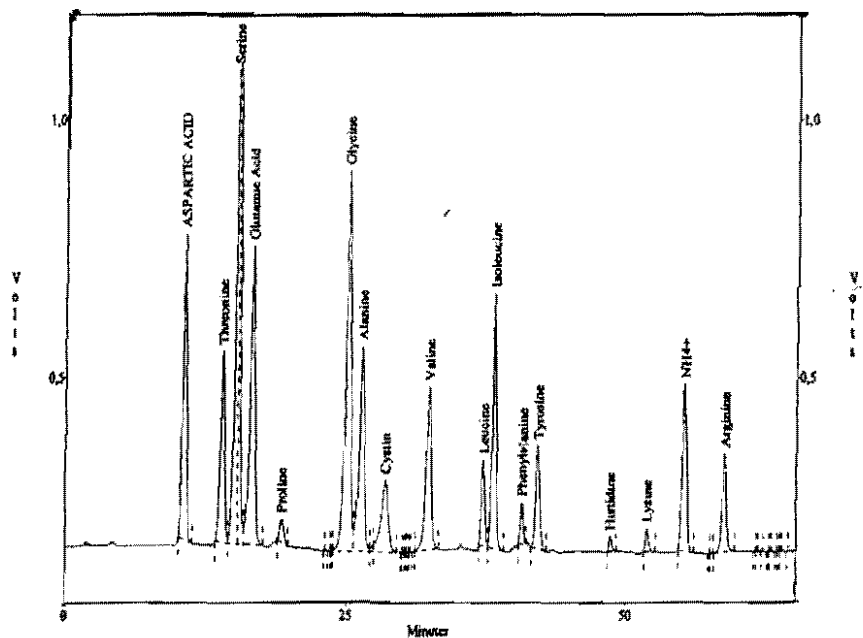


Fig. 1. Amino acid analysis of CF after hydrolysis with 6N HCl, at 110°C. for 24h.

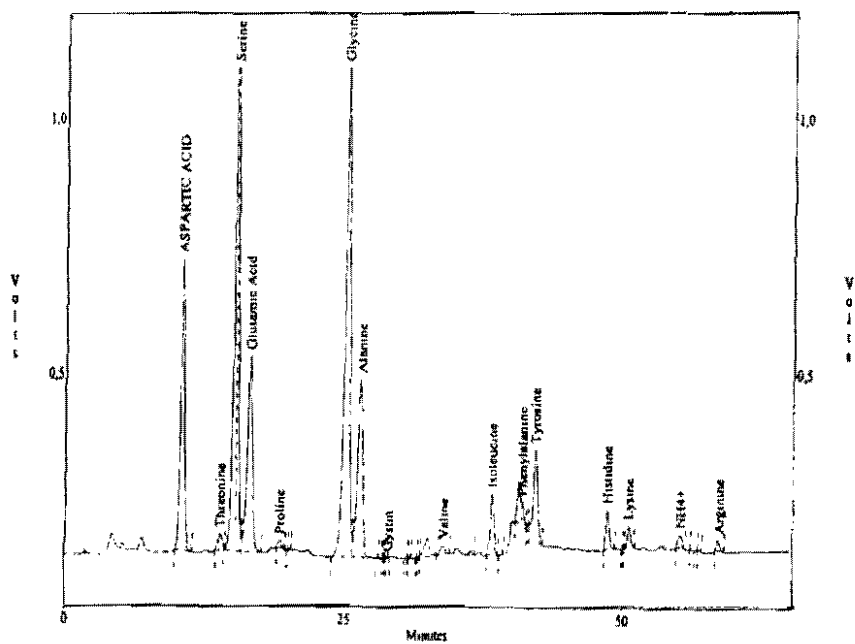


Fig. 2. Amino acid analysis of CF after hydrolysis with 0.95N NaOH, at 70°C. for 1h.

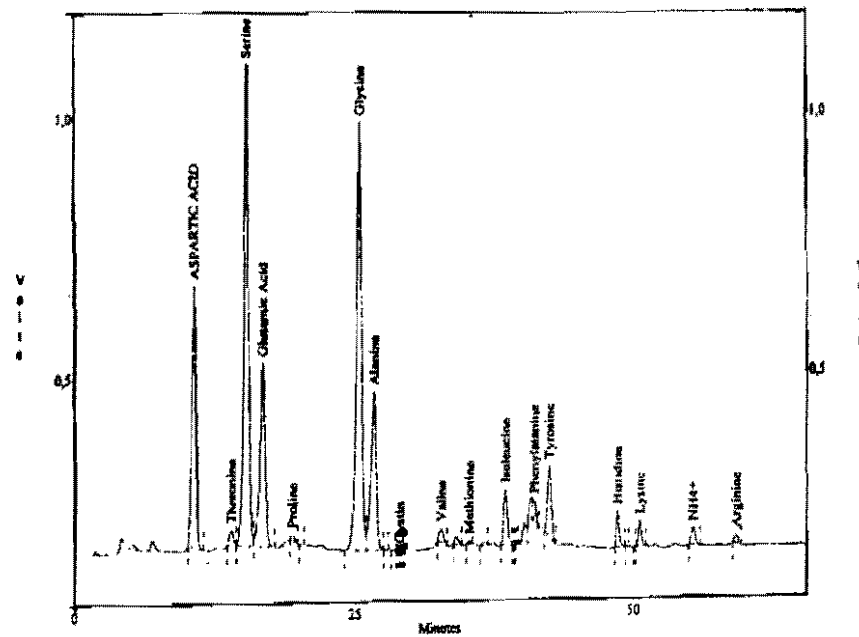


Fig. 3. Amino acid analysis of CF after hydrolysis with 0.6N NaOCl, at 80°C, for 2h.

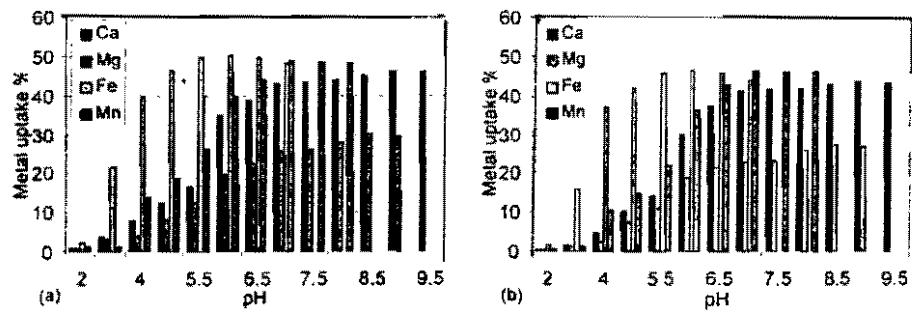


Fig. 4. Sorption behavior of metal ions on 0.2g sorbent (a) NaOH-treated CF-immob. silica, (b) NaOCl-treated CF-immob. silica, as a function of pH. Sample volume, 100 ml; concentration of each metal ion is 100 mg l⁻¹ at 25°C for 30 min.

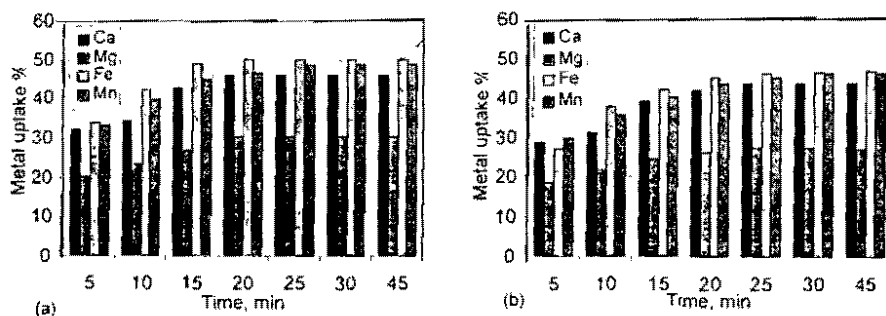


Fig. 5. Sorption behavior of metal ions on 0.2g sorbent (a) NaOH-treated CF-immob. silica, (b) NaOCl-treated CF-immob. silica, as a function of time. Sample volume, 100 ml; concentration of each metal ion is 100 mg l^{-1} at 25°C .

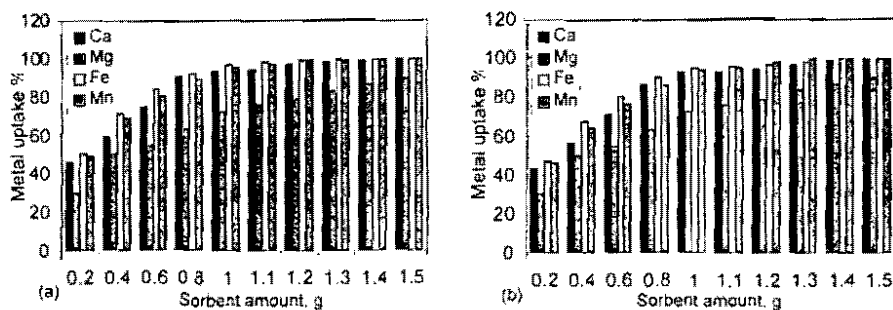


Fig. 6. Sorption behavior of metal ions on different sorbent amounts (a) NaOH-treated CF-immob. silica, (b) NaOCl-treated CF-immob. silica. Sample volume, 100 ml; concentration of each metal ion is 100 mg l^{-1} at 25°C .

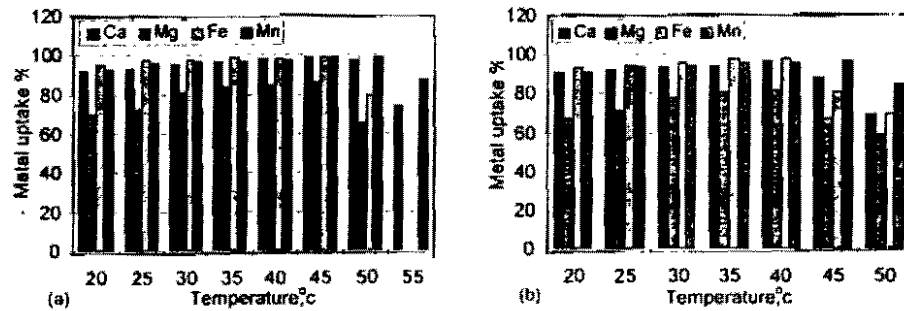


Fig. 7. Sorption behavior of metal ions on 1.0g sorbent (a) NaOH-treated CF-immob. silica (b) NaOCl-treated CF-immob. silica, as a function of temperature. Sample volume, 100 ml; concentration of each metal ion is 100 mg l^{-1} .

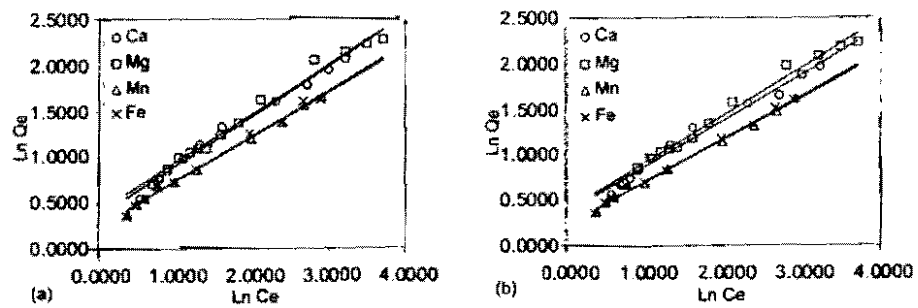


Fig. 8. Freundlich isotherms for sorption of metal ions on 1.0g sorbent (a) NaOH-treated CF-immob. silica, (b) NaOCl-treated CF-immob. silica. Sample volume, 100 ml; different concentration of each metal at 25°C .

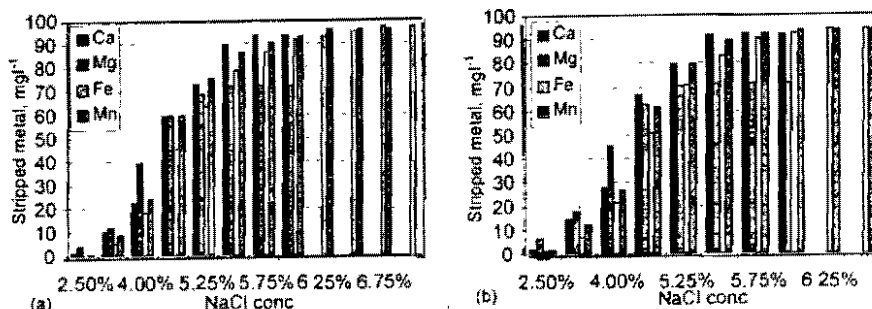


Fig. 9. Effect of NaCl concentration on stripping of asorbed metal ions from 1.0g sorbent (a) NaOH-treated CF-immob. silica, (b) NaOCl-treated CF-immob. silica. Sample volume, 10 ml at 25°C.

3. CONCLUSIONS

By alkaline treatment of chicken feathers with NaOH or NaOCl and immobilization of resultant hydrolyzates on silica, the prepared sorbents could be used to remove Ca, Mg, Fe and Mn ions contaminated water. 1.0g of NaOH-treated CF-immobilized silica could remove 93.38% Ca, 72.55% Mg, 97.13% Fe and 95.66% Mn, while, 1.0g of NaOCl-treated CF-immobilized silica could remove 92.14% Ca, 71.85% Mg, 94.26% Fe and 93.57% Mn in pH, 9, 8.5, 6 and 7, within 20-30 minutes, respectively. Metal ions uptake increased with temperature till certain temperatures, then decreased. Metal ions uptake increased little with counter anions like nitrate, bicarbonate and sulfate. However, in high concentrations of sulfate metal uptake decreased. pH played an important role in metal sorption selectivities. The sorbents could be regenerated with NaCl solution. These cheap regenerable sorbents could be applied economically to remove metals contaminated Egyptian industrial water.

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إزالة بعض الفلزات كملوثات من مياه الصرف باستخدام ريش الدجاج

يهدف هذا البحث الى إستخدام ريش الدجاج كمصدر لأحماض أمينية بعد معالجته بقلوى وتثبيتته على سطح السيلكا كمادة مازة لفلزات الكالسيوم ، الماغنسيوم ، الحديد والمنجنيز. أجريت تجارب الإمتزاز لدراسة تأثير الرقم الهيدروجيني للمحلول ، الزمن - كمية المادة المازة ، تركيز الفلز - درجة الحرارة وجود أيونات أخرى وذلك على كفاءة إمتزاز العناصر النزوية. تم مناقشة النتائج وتطبيق المعادلات الساندة فى هذا المجال.