Benha University
Faculty of Science
Chemistry Department

Synthesis, characterization and application Of Modified cellulose from lignocellulosic Waste For waste water treatment

A Thesis

Submitted in Partial Fulfillment of the Requirements for the Master Degree in Science (M. Sc).

by

Alaauddin Mohamed Ezzat El-Anwar Ahmed
B.Sc. (Chemistry)

Supervised by

Prof.Dr. Ali Hashem Mohamed
Professor of Applied Organic Chemistry
Textile Research Division,
National Research Centre

Dr.Ahmed Abd-Elsalam Khalil
Assistant Professor of Organic Chemistry
Faculty of Science, Benha University

Dr. Hesham Hafez Sokker
Assistant Professor of Applied Organic Chemistry,
National Centre for Radiation Research and Technology,
Nasr City

2009

بسم الله الرحمن الرحيم

وقل اعملوا فسيرى الله

عملكم ورسوله والمؤمنون

صدق الله العظيم

التوبه - الآيه (١٠٥)

Approval Sheet

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To Faculty of Science, Chemistry Department, Benha University

Supervisors — Approved

1. Prof. Dr. Ali Hashem ……..
Professor of Applied Organic Chemistry and Chemical Technology, Textile Research Division, National Research Centre, Dokki, Cairo

2. Dr. Ahmed Abd-Elsalam Khalil ……..
Assistant Professor of Organic Chemistry, Faculty of Science, Benha University

3. Dr. Hesham Hafez Sokker ……..
Assistant Professor of Applied Organic Chemistry, National Centre for Radiation Research and Technology, Nasr City

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Arbitrators

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Publication from the present work

Part of the present work has been subsequently published and is listed below as follows:

-"Preparation, Characterization and Utilization of Amidoximated Poly (MAA / AN)-Grafted Alhagi Residues for the Removal of Zn (II) from Aqueous Solution"

A.A.Khalil, H.H.Sokker, A.Al-Anwar, A.Abd ElZahar and A.Hashem
Adsorption science & technology, JApAn (Accepted 2009)

To My FaMily For

Sincere Help

Summary

Summary

The original work presented in this thesis was undertaken with the primary objective of synthesizing new polymeric materials based essentially on Alhagi residues to be used as adsorbent materials.

To achieve the goal, Alhagi residues was subjected to grafting reaction using MAA/AN monomer in presence of dimethyl sulfoxide as solvent and γ-irradiation as initiator.

The so-obtained grafted Alhagi residues was treated with hydroxyl amine hydrochloride in alkaline medium to obtain amidoximated poly (MAA/AN)-grafted Alhagi residues.

In short, the work comprises the following investigations

1. Estimation of cellulose content in Alhagi residues.

2. Studying all factors affecting grafting of MAA/AN on Alhagi residues.

These factors include:

-radiation dose

-monomer compositions and concentration

3. Characterization of the grafted samples using:

- FT-IR spectroscopy

-SEM

-Estimation of nitrogen content

-Estimation of carboxyl content.

4. Treatment of grafted Alhagi residues with hydroxyl amine in alkaline medium to obtain amidoximated poly (MAA/AN) –grafted Alhagi residues.

5. Characterization of amidoximated poly (MAA/AN) grafted Alhagi residues using:

-FT-IR spectroscopy

-SEM

-Estimation of percent solubility in distilled water

-Estimation of nitrogen content

-Estimation of carboxyl content

6. Utilization of the so-obtained amidoximated poly (MAA/AN) –grafted Alhagi residues for the removal of Zn (II) from aqueous solution.

Results of these investigations led to the following:

-Alhagi residues contain 5% ash, 25% lignin, 37.1 alpha cellulose and 25% hemi-cellulose and 5.9 % extractive material.

- The FT-IR spectra of grafted Alhagi residues shows the appearance of a new absorption peak (sharp peak) at 2244.25 cm^{-1}, which is attributed to the cyanide (CN) groups.

- The FT-IR spectra of amidoximated poly (MAA/AN)-grafted Alhagi residues shows disappearance of the cyanide peak and appearance of new absorption peak at 1655.73 cm^{-1} corresponding to the stretching vibration of the C=N bond which indicate the conversion of the original cyanide groups to amidoxime groups.

-The graft yield % increase by increasing radiation dose from 5 to 10 kGy and then remained constant at higher radiation doses (15-20 kGy).

- The graft yield % increase by increasing the comonomer composition up to 60/40 (MAA/AN) and then decreases.

- The appropriate condition for grafting of MAA/AN Alhagi residues could be achieved using radiation dose of 10kGy and MAA/AN ratio of 60-40.

The adsorption data indicate that the amidoximated samples were effectively used in adsorption of Zn (II) rather than Hg (II) and Pb (II) from aqueous solutions.

-The adsorption capacity of Zn (II) onto amidoximated poly (MAA/AN) – grafted Alhagi residues were 212.76 and 344.8 mg/g at 30 and 50°C, respectively.

-The adsorption data obeyed both Langmuir and Freundlich isotherms and followed second order kinetics.

CONTENTS

CHAPTER II(Experimental)

CHAPTER III (Results and Discussion)

Appendix

Symbol	Signification
AR	Alhagi Residues
AN	Acrylonitrile monomer
MAA	Methacrylic Acid
GAR	Grafted Alhagi Residues
AAR	Amidoximated Alhagi Residues
C_o	Initial concentration of adsorbate in solution (mg/l)
C_e	Equilibrium liquid phase concentration (mg/l)
q_e	Amount of adsorbed metal at equilibrium (metal uptake), mg of metal per gram of adsorbent
q_t	Amount of adsorbed metal at time t (mg/g)
W	Weight of adsorbent (g)
W_o	Weight of Alhagi residues before grafting,
W_g	Weight of Alhagi residues after grafting,
V	Volume of metal ion (l)
T	Time (min)
k_1	Rate constant of pseudo first-order of adsorption (min^{-1})
k_2	Rate constant of pseudo second-order of adsorption (g/mg.min)
k_p	Intra-particle diffusion rate constant (mg/g. min)
C	Constant that gives idea about the thickness of boundary layer
k_L	Langmuir constant (l/g)
a_L	Langmuir isotherm constant (l/mg)
b	Energy of adsorption (l/mg)
Q_{max}	Maximum adsorption capacity (mg/g)
R_L	Dimensionless constant separation factor parameter
K_F	Constant of Freundlich indicates the adsorption capacity

1/n	Arbitrary constant related to the adsorption intensity and favorability of adsorption
R^2	Regression correlation coefficient
R	Universal gas constant (8.31441 J/mol K)
T	Absolute temperature (K)
ΔG°	Standard free energy of adsorption (kJ/mol)
ΔH°	Standard enthalpy of adsorption (kJ/mol)
ΔS°	Standard entropy of adsorption (kJ/mol K)