THE SYNTHESIS OF CALIX[4]RESORCINARENE FROM CASSIA OIL AND ITS APPLICATION FOR SOLID PHASE EXTRACTION OF HEAVY METALS Hg(II) AND Pb(II)

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INTRODUCTION

SIGNIFICANT CONTENT OF Pb(II) AND Hg(II) ON STREAM AND INDUSTRIAL EFFLUENT MUST BE REMOVED

SOLID PHASE EXTRACTION IS ONE OF THE FEW PROMISING TECHNOLOGY TO TREAT INDUSTRIAL WASTE

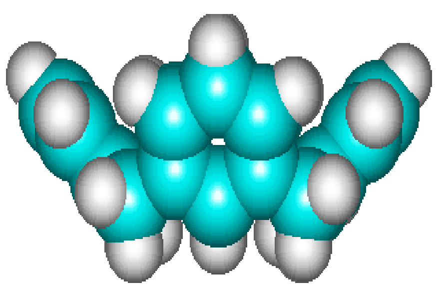
CYCLIC OLIGOMER CALIXARENES HAVE POTENT AS SOLID PHASE EXTRACTOR

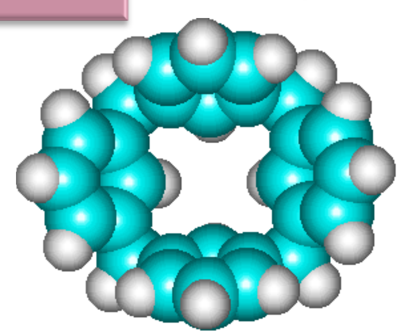
CALIXARENE

CALIXARENES ARE SYNTHETIC CYCLIC OLIGOMERS OF AROMATIC RESIDUES LINKED BY A BRIDGE.

THIS MACROMOLECULE HAS ALMOST UNLIMITED POSSIBILITIES OF MODIFICATION, INCLUDING THE MODIFICATION OF TYPE AND NUMBER OF AROMATIC RESIDUES, FUNCTIONAL GROUPS, AND BRIDGES

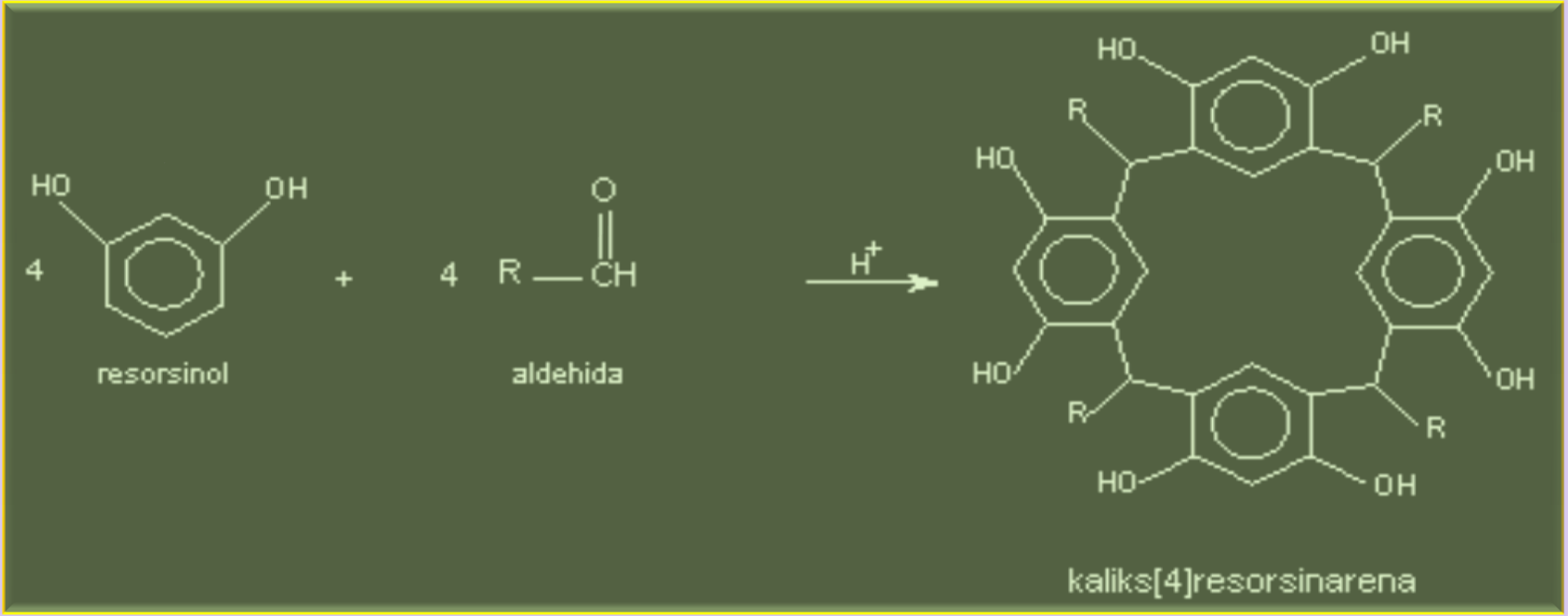
THIS FAMILY REPRESENTS AN INTERESTING GEOMETRY THAT EXHIBITS CHARACTERISTIC OF CAVITY OR BASKET SHAPE.





THIS SHAPE ALLOWS CALIXARENES APPLICATION IN HOST-GUEST SYSTEM. THE FAMILY OF CALIXARENE HAS BEEN USED FOR VARIOUS UTILITIES.

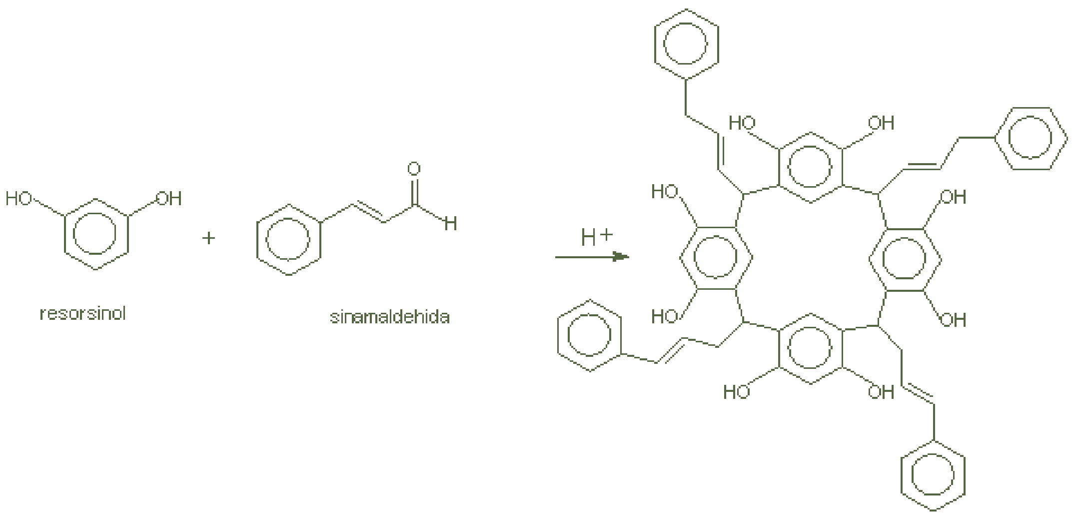
GENERAL SCHEME OF SYNTHESIS CALIX[4]RESORCINARENE



SYNTHESIS OF C-CINNAMAL CALIX[4]RESORCINARENE (CCCR) FROM CASSIA OIL

CASSIA OR CINNAMON BURMANNI, ONE OF INDONESIAN NATURAL PRODUCT

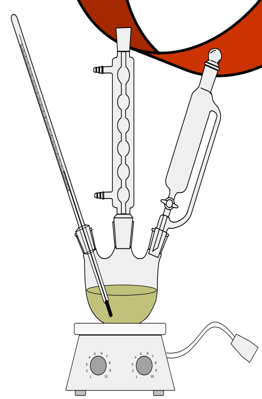
CINNAMALDEHYDE CONTENT OF CASSIA OIL IS 90%



C-sinamal Kaliks[4]resorsinarena

METHODS

- ISOLATION OF CINNAMALDEHYDE FROM CASSIA OIL
- SYNTHESIS OF CCCR
- STRUCTURE DETERMINATION OF CCCR
- SCALE UP OF CCCR

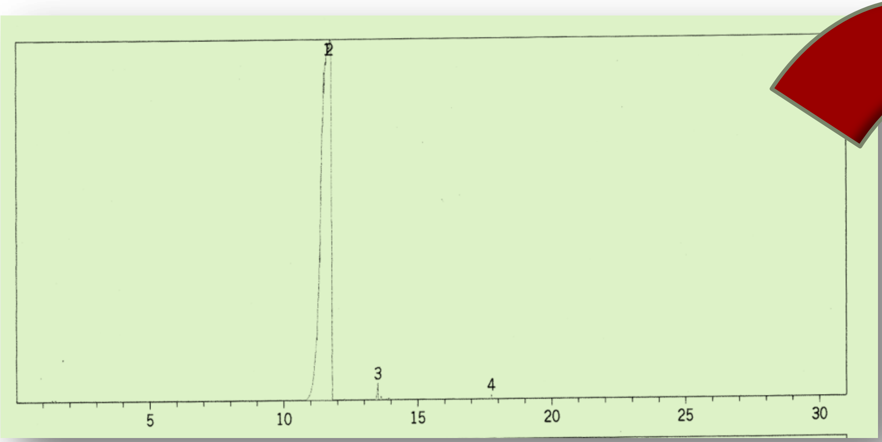


SOLID PHASE EXTRACTION

- SOLID PHASE EXTRACTION OF Pb(II)
  - EFFECT OF ACIDITY LEVEL
  - EFFECT OF CONTACT TIME → EXTRACTION KINETICS
  - EFFECT OF METAL CONCENTRATION → EXTRACTION ISOTHERM
- SOLID PHASE EXTRACTION OF Hg(II)
  - EFFECT OF ACIDITY LEVEL
  - EFFECT OF CONTACT TIME → EXTRACTION KINETICS
  - EFFECT OF METAL CONCENTRATION → EXTRACTION ISOTHERM

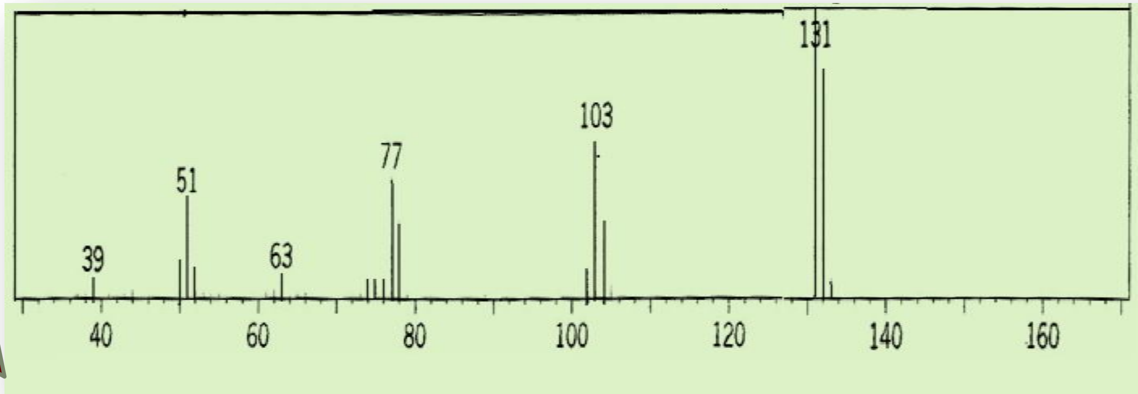
RESULTS

ISOLATION CINNAMALDEHYDE FROM CASSIA OIL



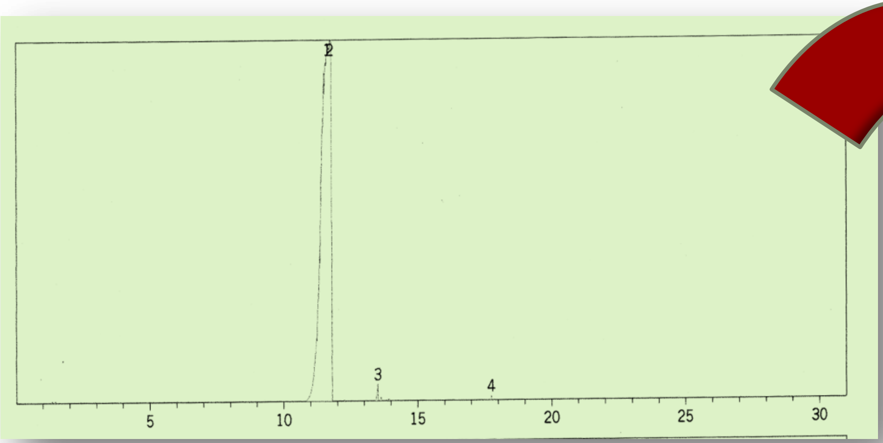
GAS CHROMATOGRAM OF CINNAMALDEHYDE SHOWS 99.5% PURITY OF ISOLATION PRODUCT

MASS SPECTRUM OF CINNAMALDEHYDE SHOWS M^+ AT $m/z = 132$



RESULTS

ISOLATION CINNAMALDEHYDE FROM CASSIA OIL



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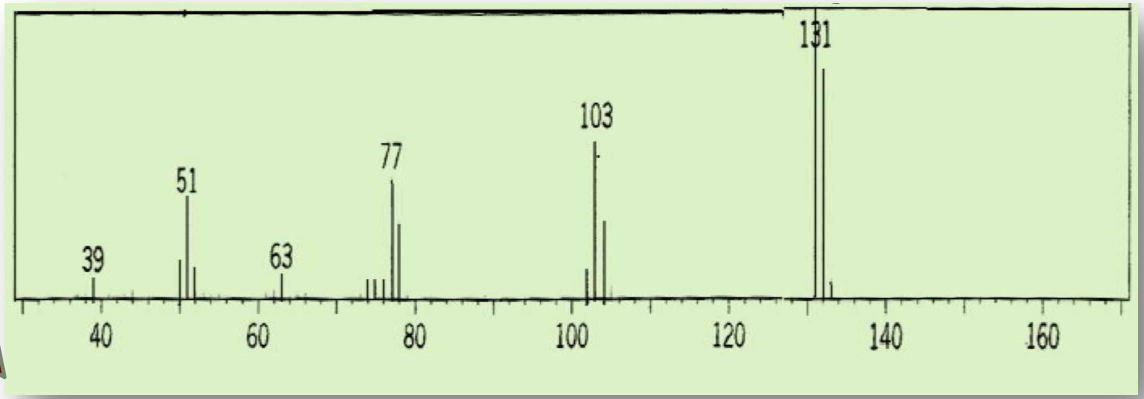
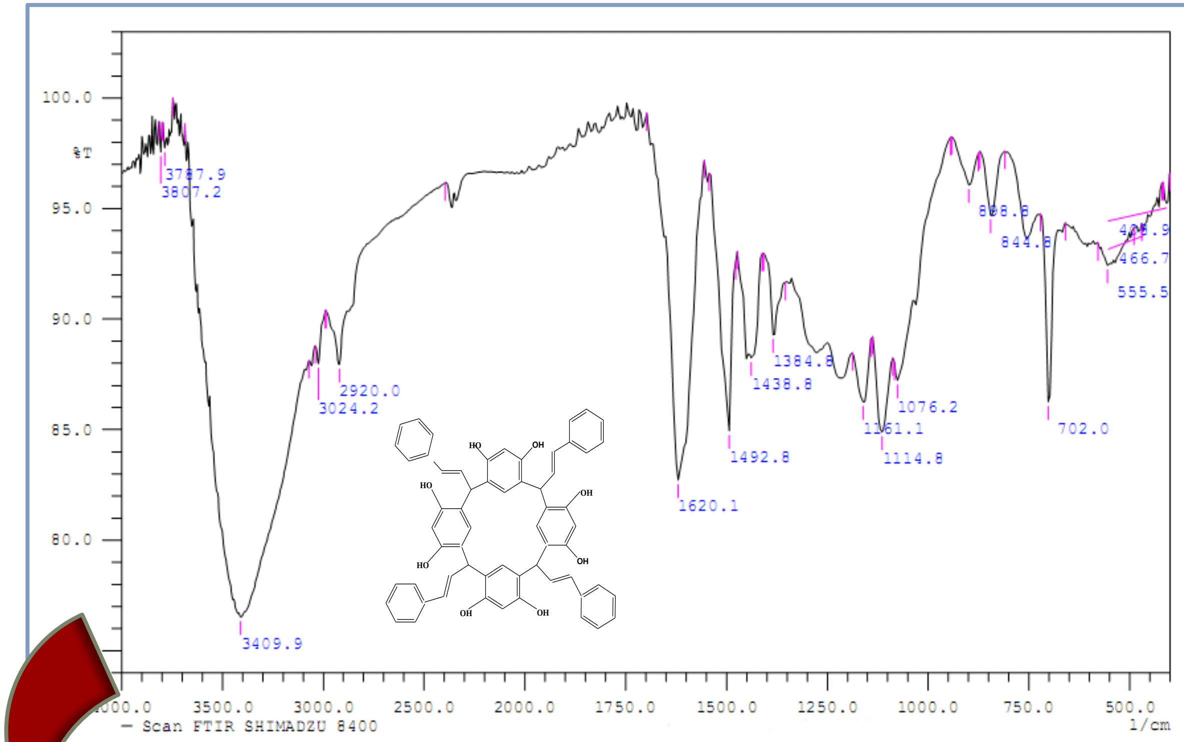


SYNTHESIS OF CCCR

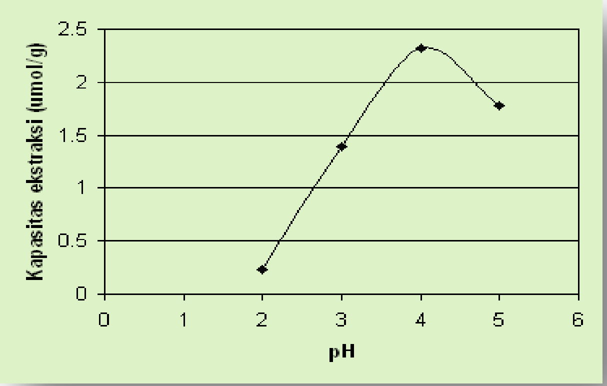


- FTIR SPECTRUM OF CCCR CONSISTENT WITH CCCR STRUCTURE
- REACTION CONDITION: 77ºC, 24 h
- PERCENT PRODUCT : 75%

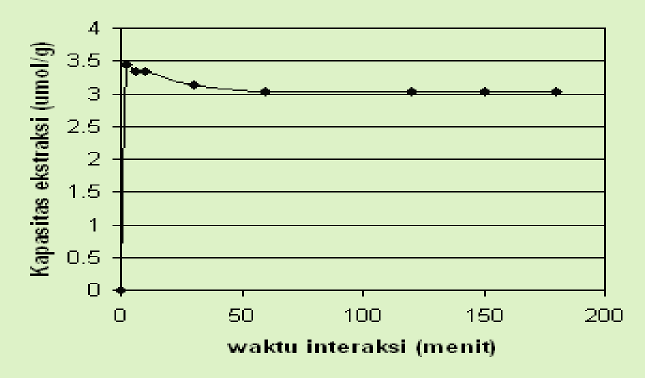
RESULTS

SOLID PHASE EXTRACTION OF Pb(II)

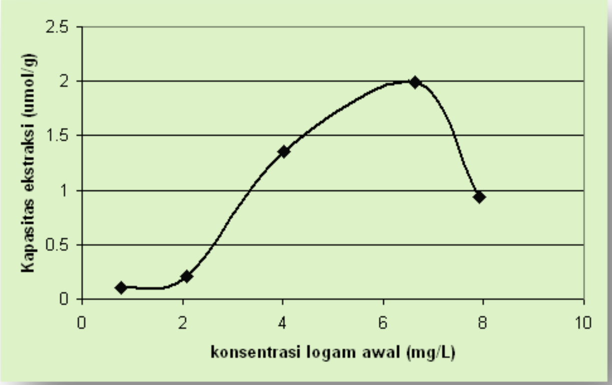
- Effect of pH



- Effect of Contact Time



- Effect of Initial Pb(II) Concentration

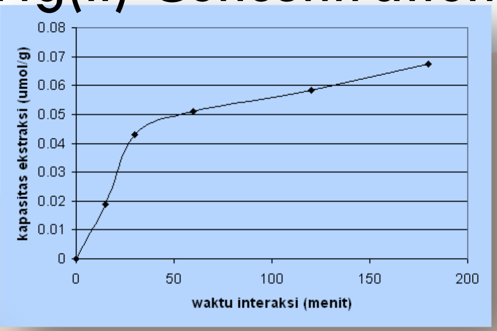


SOLID PHASE EXTRACTION OF Pb(II) WENT OPTIMAL ON pH 4, 180 MINUTES OF CONTACT TIME, AND 6.6 mg/L OF INITIAL Pb(II) CONCENTRATIONS

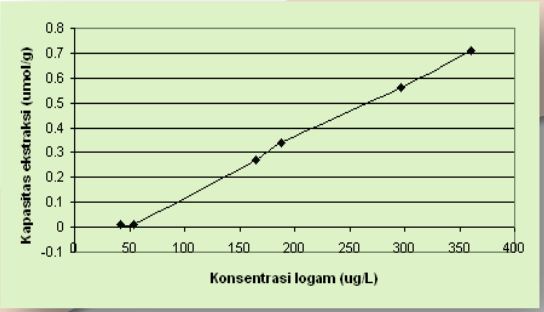
RESULTS

SOLID PHASE EXTRACTION OF Hg(II)

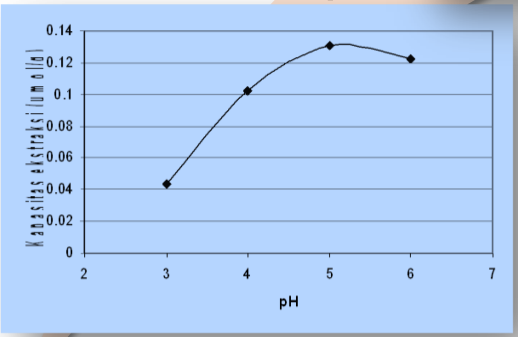
- Effect of Hg(II) Concentration



- Effect of Contact Time



- Effect of pH



SOLID PHASE EXTRACTION OF Hg(II) GAVE OPTIMAL CONDITION, i.e. PH 4, CONTACT TIME WAS 180 MINUTES, AND INITIAL Hg(II) CONCENTRATIONS WAS 0.36 mg/L.

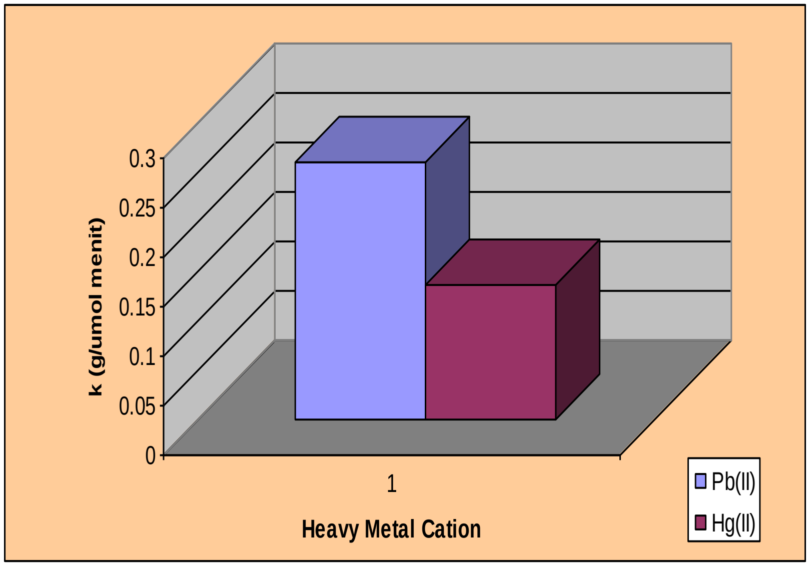
RESULTS

COMPARISON OF Pb(II) AND Hg(II) SOLID PHASE EXTRACTION

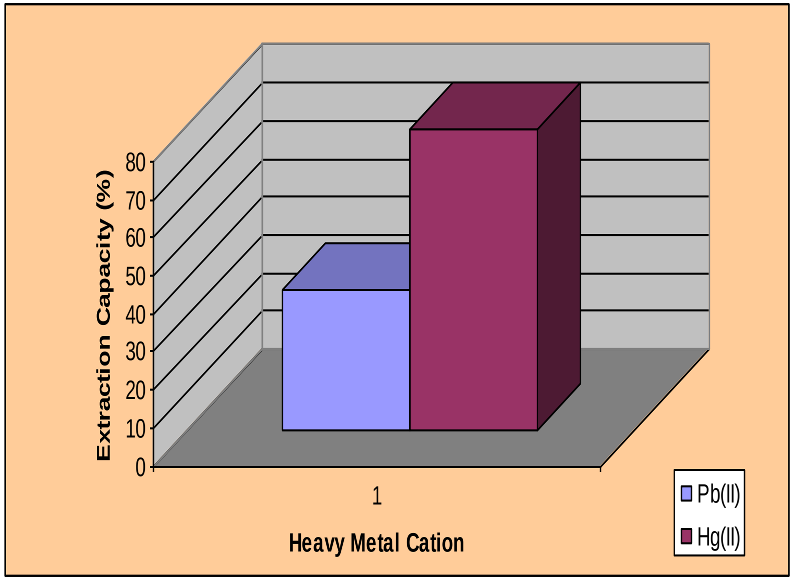
PARAMETER	Pb(II)	Hg(II)
pH OPTIMUM	4	5
OPTIMUM CONTACT TIME (MINUTE)	180	180
OPTIMUM METAL CONCENTRATION (mg/L)	6.6	0.36
KINETICS MODEL	PSEUDO 2^{nd} ORDER	PSEUDO 2^{nd} ORDER
ISOTERM MODEL	LANGMUIR	FREUNDLICH

RESULTS

COMPARISON OF Pb(II) AND Hg(II) SOLID PHASE EXTRACTION



RATE EXTRACTION Pb(II) > Hg(II) INDICATES RATE EXTRACTION WAS AFFECTED THE EXIXTENCE OF HYDROXYL GROUP, WHICH WAS STRONG ELECTRON DONATING (STRONG BASE)



EXTRACTION CAPACITY Pb(II) < Hg(II) INDICATES EXTRACTION CAPACITY DETERMINED BY THE SUITABILITY OF HARD-SOFT ACID-BASE CHARACTER

CONCLUSIONS

- CCCR PRODUCED FROM CASSIA OIL COULD BE SOLID PHASE EXTRACTOR OF Hg(II) AND Pb(II).
- SOLID PHASE EXTRACTION OF Pb(II) WENT OPTIMAL ON pH 4, 180 MINUTES OF CONTACT TIME, AND 6.6 mg/L OF INITIAL Pb(II) CONCENTRATIONS, FOLLOWED PSEUDO SECOND ORDER KINETICS MODELS, FIT WELL WITH LANGMUIR ISOTHERM MODELS, AND GAVE EXTRACTION CAPACITY IN 1.986 μMOL/g OR 37.2%.
- SOLID PHASE EXTRACTION OF Hg(II) GIVE OPTIMAL CONDITION, i.e. pH 4, CONTACT TIME WAS 180 MINUTES, AND INITIAL Hg(II) CONCENTRATIONS WAS 0.36 mg/L.
- Hg(II) EXTRACTION FOLLOWED PSEUDO SECOND ORDER KINETICS MODELS, FIT WELL WITH FREUNDLICH ISOTHERM MODELS, AND HAVE EXTRACTION CAPACITY IN 0.71 μMOL/g OR 79.1%.

THANK YOU

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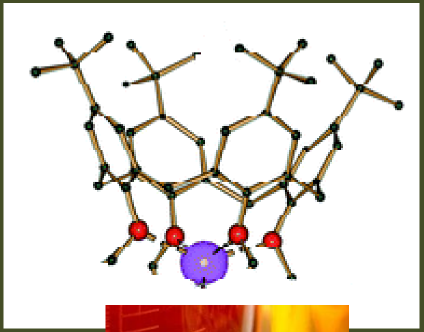
PERSAMAAN MODEL KINETIKA DAN ISOTERM EKSTRAKSI FASA PADAT

Model kinetika ekstraksi Pb(II) dan Hg(II) oleh CSKR

Model Kinetika	R^2	
	Pb(II)	Hg(II)
Persamaan pseudo orde 1 (Lagergren) $\log(q_e - q) = \log q_e - (k/2.303)t$	0.6552	0,9076
Persamaan pseudo orde 2 (Ho) $t/q = ½\, kq_e^2 + t/q_e$	0.9999	0,9817



Model isoterm ekstraksi Pb(II) dan Hg(II) oleh CSKR

Model Isotherm	**Hg(II)**		**Pb(II)**	
	Persamaan linier	R^2	**Persamaan linier**	R^2
Freundlich	y=3,3901x+1,286	0,9949	y=0,5708x-0,795	0,6640
Langmuir	y=2,679x-5,7328	0,9999	y=2,8208x+2,6527	0,9023