UNIVERSITI TEKNOLOGI MARA

OPTIMIZATION OF HEAVY METALS REMOVAL FROM AQUEOUS SOLUTION USING MAGNESIUM-ALUMINIUM NITRATE AND INTERCALATED MAGNESIUM-ALUMINIUM-TARTRATE LAYERED DOUBLE HYDROXIDES

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AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and the result of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution of any degree or qualification.

I hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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ABSTRACT

Mg-Al-NO_3 and intercalated Mg-Al-tartrate layered double hydroxides were synthesized by co-precipitation method for the removal of lead, cadmium and copper from aqueous solutions. The characterization of Mg-Al-NO_3 by using XRD analysis showed the presence of sharp peaks which signifying high crystallinity. The presence of sharp and intense lines with d-spacing of 7.95 Å demonstrated general features of layered double hydroxides. The basal spacing for Mg-Al-tartrate was recorded at 8.14 Å which indicates larger d-spacing as compared to the Mg-Al-NO_3. The FTIR analysis showed the strong absorption band was recorded at 1631 $cm^{-1}$for Mg-Al-NO_3-tartrate which attributed to the symmetric and asymmetric vibration of coordinated $-COO^-$ group from organic acids. The two stretching vibrations of the alcoholic groups in tartrate are centered at 1111 cm^{-1} and 1066 cm^{-1} which recorded at 1140 cm^{-1} and 1082 cm^{-1} for the intercalated spectrum. The effects of various experimental parameters on the removal of heavy metals such as contact time, different concentration, pH solution, and adsorbent dosage were investigated by batch method at temperature of 25°C. The percentage removal of heavy metal ions for each of the parameters studied was determined by ICP-OES. The extent of heavy metals ions removal increased with the increased in contact time and the dosage of LDHs used. The higher percentage removal by Mg-Al-tartrate is mainly due to the function of organic anion in the layer which possibly forms complexes with heavy metals. The adsorption experiments of heavy metal solution with high acidity or alkalinity are not desirable in which at high acidity the LDHs will dissolve and at alkaline condition the heavy metals ions are likely to form insoluble precipitates with NaOH. Kinetic study showed that the adsorption of heavy metal ions on LDHs was a gradual process and pseudo second-order rate equation was able to provide realistic description of adsorption kinetics based on equilibrium loading value of experiment (q_{exp}). The shape of the isotherms that obtained from the experimental data was recorded well fitted to the Langmuir isotherm with regression coefficient near to one. The RSM based on a three and four-level CCRD was employed to evaluate the interactive effect of the various optimization parameters. The experimental percentage recorded under optimum conditions was compared well with the maximum predicted value from the RSM with not more than 1% of differences, which suggest that CCRD of RSM can be used to study the removal of heavy metal ions from aqueous solution.

TABLE OF CONTENTS

CHAPTER ONE
INTRODUCTION

1.1 DISSERTATION OUTLINE

Recently, layered double hydroxides (LDHs) have gained an interest in a wide variety of industries, as well as in research. A number of researches have been carried out on the pollutant removal of wastewater by using LDHs. LDHs have the ability to treat wastewater with higher percentage removal of the pollutant. In this study, non-intercalated Mg-Al-NO_3 and intercalated Mg-Al-tartrate LDHs was used as an adsorbent to remove heavy metal ions from aqueous solution.

Chapter One explained the layered double hydroxides and their historical background. The environmental problems that occurred contribute to the further research and enhancement application of layered double hydroxides also discussed in this chapter. This chapter also affirmed the objectives, significances, scopes and limitation of the study.

Chapter Two presents the details review of the literature for this study. This chapter focused on previous repeated research that provide the knowledge of methods or techniques used in synthesis and characterize the structure of layered double hydroxides and related study with the adsorption processes.

Chapter Three described the details about experimental work that was carried out. Starting with the chemical used, method of synthesis, characterization with selected instruments, conventional adsorption experiment and finally the optimization by used of Response Surface Methodology (RSM).

Chapter Four presents the results and discussion. This chapter covers the details about results of layered double hydroxides characterization before and after adsorption experiments and also adsorption experiment according to the parameter study, kinetic and isotherm of adsorption and finally the result of optimization by used of Response Surface Methodology.

Chapter Five points of summarized and conclusion for the study and point of recommendation that can be used to improve the study of layered double hydroxides in other fields of research.