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Original

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Summary

This work is focused on the investigation of innovative materials for CO₂ capture from flue gas streams in a post-combustion carbon capture process. Porous solid sorbents and bio-based ionic liquids are applied to accomplish the objective of our study by adapting and modifying their properties, which contribute to a higher system efficacy and lower energy requirement. The results obtained from solid sorbent performance are modeled for a Temperature Swing Adsorption (TSA) system and validated using the experimental results.

In **Chapter 1** of this thesis, the main technologies developed for CO₂ removal from flue gases along with their pros and cons are described. Different types of materials used in these technologies are discussed with the main focus on the application of porous solid sorbents including a natural zeolite known as Clinoptilolite (Clino) and bio-based Ionic liquid famous as Amino-acid Ionic Liquid ([AA][IL]).

In **Chapter 2** the application of clinoptilolite for CO₂ separation from industrial processes at moderate temperatures in the range of 50 °C - 100 °C is studied in detail. Besides evaluating the sorbent in its raw form, the properties and the adsorption performance of cation-exchanged modified clinoptilolite are also investigated. The capability of zeolites in exchanging cations makes them a unique and potential sorbent with a basic structure for CO₂ separation purposes. In this work, the effect of alkali (Na⁺) and alkaline metal (Ca²⁺) cations on adsorption features are evaluated. Several tests are performed to understand the kinetic and equilibrium behavior of the sorbent as well as its CO₂ uptake performance and efficiency. The sample's characterization is done by means of EDX, XRD and N₂ physisorption analysis giving insight into the chemical composition and porous structure of the sorbent. Moreover, the adsorption isotherm for pure CO₂ at different temperatures is measured with the volumetric method. Using the results obtained from adsorption isotherms at two different temperatures, the isosteric heat of adsorption is evaluated. The adsorption isosteric heat profile verified the presence of strong sites on the structure of the zeolite leading to the formation of different species. To understand the nature of these species contributing to the isosteric heat of adsorption the IR spectroscopy analysis is carried out. The results obtained from this comprehensive study indicated the high thermal stability of untreated clinoptilolite which causes an increase in the sorbent CO₂ loading uptake. In perspective, the improved adsorption capacity of clinoptilolite, versus the state-of-the-art zeolite 13X, at high temperature makes it a potential sorbent for CO₂ removal from flue gases, with temperature ranges between 80°C and 100 °C.

In **Chapter 3** the zero-length column (ZLC) method for measuring the diffusion and transport mechanism of CO₂ into the sorbent is described in detail. Later the method is used to evaluate the mass transfer behavior for Clino, K-Clino, Na-Clino and Cs-Clino. The results obtained confirmed that the nature of CO₂ adsorption on Clino and its cation exchanged forms is equilibrium controlled.

Alternatively, the sorbent adsorption capacity is evaluated by measuring the CO₂ equilibrium isotherms and adsorption breakthrough curves. The adsorption robusticity of the sorbent is examined by repeating the adsorption (20 °C, 35 °C, 50 °C and 65 °C) and desorption (100 °C and 300 °C) at different temperatures and through different cycles.

Chapter 4 discusses a mathematical model developed for a one-column fixed-bed reactor to investigate the dynamic behavior of CO₂ adsorption process. The model is validated with the experimental data obtained from the same system using commercial zeolite 13X. The process is considered to work in three stages of adsorption, desorption, and cooling. Temperature Swing Adsorption (TSA) configuration is devoted to the regeneration phase by heating the system up to 85 °C. The operability of each phase is exploited through a one-dimensional model using mass, energy and momentum balances. The input parameters for equilibrium data are obtained by conducting equilibrium experiments for pure component and by fitting the data with single and dual-site Langmuir isotherm. The parameters obtained from the adsorption equilibrium model is used to investigate the kinetic parameters necessary for building the numerical model.

Chapter 5 is dedicated to the absorption study carried out by using amino acid-based ionic liquids [AA][IL] with a lower environmental impact and energy demand comparing to conventional solvents (e.g. amines). Four choline-based amino acids ([Cho][AA]): Alanine, Glycine, Proline, and Serine, that are task-specific for this purpose are tested. The drawbacks related to the high viscosity of these ILs were limited by applying DMSO as a solvent, which is chosen because it is a polar aprotic liquid with low toxicity, low vapor pressure, and relatively low price. The absorption measurements are performed at constant operational conditions of 20 °C (using circulating thermal bath) and 1 bar. The optimal concentration of IL in DMSO (12.5 and 25 wt%) is examined through preliminary tests performed with various concentration of DMSO. The solution of these ionic liquids in DMSO showed promising properties for CO₂ removal from a simulated flue gas mixture with 17 vol% CO₂ in Ar. Indeed, the higher absorption efficiency is the outcome of diluting the IL in the DMSO, which contributes to minimize the CO₂ mass transfer limitation toward the ILs absorption. These solutions are potentially competitive due to their low cost, low environmental impact, easy processability (due to their low viscosity) and good regenerability for the production and storage of highly pure CO₂.

Finally, in **Chapter 6** the principle findings of this dissertation to create a cost-effective process through application of Clinoptilolite zeolite and Bio-based ionic liquids are discussed.