

X型沸石分子筛吸附剂的制备及其吸附性能研究

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Preparation and Adsorption Performance of X Zeolite Adsorbent

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论文单位 太原理工大学,点击次数 30,论文页数 66页File Size3223K

2007-05-01 [论文网](http://www.lw23.com/lunwen_620127447/) http://www.lw23.com/lunwen_620127447/

Zeolite;; adsorption property;; binder;; alkali treatment;; modification

变压吸附分离技术(PSA)是近年来在分离工业新崛起的气体分离技术,其原理是利用气体组分在固体材料上吸附特性的差异以及吸附量随压力变化而变化的特性,通过周期性的压力变换过程实现气体的分离或提纯。而吸附剂是变压吸附分离技术(PSA)的基础,吸附剂的性能直接影响最终分离效果,甚至影响工艺步骤的复杂性和吸附操作单元的使用寿命。因此,在PSA技术中对吸附剂的研究极为活跃。本课题以提高沸石分子筛吸附剂的吸附性能为目的,考察了X型沸石分子筛成型过程中粘结剂的选择、焙烧温度的选择,碱溶液处理对型体分子筛吸附性能的影响以及粉体X型沸石分子筛改性研究。并以此为依据制备出了具有高吸附容量沸石分子筛吸附剂。以凹凸棒土为粘结剂制备型体13X沸石分子筛(13X/A),对其吸附性能进行了深入研究,并与以高岭土为粘结剂的型体13X沸石分子筛(13X/K)进行比较。研究结果表明凹凸棒土凭借自身丰富的孔道结构所赋予的较大的比表面积、孔体积,在沸石分子筛成型过程中不仅起到粘结的作用,而且还提高了型体13X沸石分子筛的吸附容量。在所考察的焙烧温度下,随焙烧温度升高型体13X/A沸石分子筛对CO₂、H₂O的平衡吸附量变化规律与凹凸棒土的比表面和孔体积在此焙烧过程中的变化规律是一致的。在823.15K时,型体13X/A沸石分子筛呈现出最高的吸附容量,其对CO₂、N₂、CO和CH₄的平衡吸附量分别为105.80ml/g、5.93ml/g、12.34 ml/g和10.59 ml/g,较型体13X/K沸石分子筛,分别提高了2.95 ml/g、0.19ml/g、2.06ml/g和1.55 ml/g,表现出更高的吸附容量。用碱处理方法对型体13X沸石分子筛进行处理,旨在提高其吸附性能。研究结果表明,碱溶液处理可以显著提高型体13X沸石分子筛的吸附性能。比较可以发现,碱处理后型体13X/K沸石分子筛的吸附容量提高幅度要大于型体13X/A沸石分子筛,从而表现出更优的吸附性能。碱处理前后型体13X沸石分子筛结构的变化通过XRD和低温N₂吸附-脱附进行了表征,并且对其碱处理的作用机理进行了初步探讨。研究结果表明,碱处理可以提高型体13X沸石分子筛的相对结晶度,从而提高了其比表面积和孔体积,使其对CO₂、N₂、CO和CH₄等吸附质的平衡吸附量得以提高。型体13X/A沸石分子筛结晶度的提高,比表面积和孔体积的增大可归因于其中无定形杂质在碱处理过程中的溶解;而在碱处理过程中型体13X/K沸石分子筛中的部分高岭土转变成分子筛则是其比表面积和孔体积增大的原因。为进一步提高X型沸石分子筛的吸附性能,本文对其进行了改性研究,旨在降低硅铝比,增加其骨架内的铝含量,提高其比表面积和微孔体积,由此提高其吸附性能。从而为型体13X沸石分子筛吸附剂改性制定出最佳方案,制备出更高吸附性能的吸附剂。研究结果表明,改性可以显著提高X型沸石分子筛对CO₂、N₂和CO的平衡吸附量。经改性后样品的比表面积、微孔体积均较处理前有大幅度提高,较大的比表面积、孔体积使得处理后得样品的吸附性能得到改善。

Pressure swing adsorption (PSA) is a new gas separation technology developed in the separation industry recently. PSA is well suited to the removal and subsequent recovery of CO₂ from gases due to its ease of applicability over a relatively wide range of temperature and pressure conditions and its energy efficiency. its principle is a cyclic batch process where adsorption is carried out at a relatively higher pressure and desorption (regeneration) is accomplished at a lower pressure, generally using part of the product from the adsorption step. But a key aspect in the separating is the identification of a suitable adsorbent, and the performance of adsorbent influenced directly the effect of separation. In order to improve the adsorption performance of traditional zeolite adsorbent, the influence of binder in Shaping, calcination temperature, alkali treatment on the structure and adsorption property was investigated using CO₂, N₂, CO and CH₄ as probe. The result showed that after alkali treatment both of the two shaped 13X zeolite (shaped 13X/A zeolite and shaped 13X/K zeolite) exhibit higher adsorption capacity for CO₂, N₂, CO and CH₄, And the adsorption capacity of CO₂, N₂, CO and CH₄ on shaped 13X/K zeolite are higher than that of shaped 13X/A zeolite. The influence of NaOH treatment on the crystal and pore structure was examined by XRD and N₂ adsorption-desorption (77K), and The possible mechanism for alkali treatment was also discussed. Results present that after the treatment of alkali solution, shaped 13X/A zeolite exhibits higher crystallinity, larger micropore surface area and volume compared with the origin sample. And such change improves the adsorption capacity of shaped 13X/A zeolite for CO₂, N₂, CO and CH₄ evidently. The dissolution of some amorphous species in shaped 13X/A zeolite into NaOH solution may be responsible for the higher crystallinity and larger surface area of treated shaped 13X/A zeolite. However, treated shaped 13X/K zeolite not only shows even more higher crystallinity compared to the starting sample, but also have some characteristic XRD peak of zeolite A, the results indicate that some kaolin in shaped 13X/K zeolite transform into zeolite in the process of alkali treatment, and this may be responsible for the higher crystallinity and larger surface area of treated shaped 13X/K zeolite. And such changes improve the adsorption capacity of the shaped 13X/K zeolite for CO₂, N₂, CO and CH₄ adsorption properties of shaped zeolite X adsorbent, and modification of zeolite X were investigated. Zeolite 13X were shaped using attapulgite (13X/A), the adsorption properties of the samples were investigated. And the adsorption performance of shaped 13X zeolite with attapulgite and that with kaolin(13X/K) was also compared. Results show that owing to the unique pore structure and large surface area, attapulgite has additional effect of improving adsorption capacity of shaped 13X/A zeolite in addition to hindering. In the range of calcination temperature investigated, the surface area and pore volume of shaped 13X/A zeolite reach the optimum value at 823.15K, and then decrease with the further increase of temperature. According to the change of surface area and pore volume, the shaped 13X/A zeolite calcined at 823.15K also show the largest adsorption capacity for CO₂ and H₂O. when calcined at 823.15K, shaped 13X/A zeolite have higher adsorption capacities. The adsorption capacity of shaped 13X/A zeolite for CO₂, N₂, CO and CH₄ is 105.80ml/g, 5.93ml/g, 12.34 ml/g and 10.59 ml/g respectively, and enhanced 2.95 ml/g, 0.19ml/g, 2.06ml/g and 1.55 ml/g comparing with shaped 13X/K zeolite, exhibiting the higher adsorption capacity. In the purpose of enhancing its adsorption performance, the two kinds of shaped 13X zeolite (shaped 13X/A zeolite and shaped 13X/K zeolite) were treated by certain concentration of NaOH solution. The influence on evidently. For further enhancing adsorption performance of X zeolite, in this paper, zeolite X were desiccated with aqueous solutions of NaOH and Na₂CO₃, and also were realuminated with aqueous solutions of NaOH and Na₂CO₃ and NaAlO₂. The products were characterized by using X-ray diffraction, N₂ adsorption-desorption (77K) and Fourier Transformer Infrared Spectroscopy et al. After thus treatment, the influence on adsorption property were also investigated using CO₂、N₂ and CO. The result showed that after thus treatment zeolite X exhibit higher adsorption capacity for CO₂, N₂ and CO. and larger micropore surface area and volume compared with the starting sample.

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