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Effect of operating conditions on the hydrophobisation of silica-based porous particles in a fluidised-bed reactor: Temperature effect

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ABSTRACT

This study concerns the modification of silica surface wettability by silanisation reaction which consists in the transformation of hydrophilic silanol groups into hydrophobic groups. The chemical modification of silica surface is carried out using *n*-Octadecyltrichlorosilane (ODTCS). It is a reagent with three chlorines as functional group and with one long alkyl chain containing 18 carbon atoms, responsible for the hydrophobic behavior. The reaction is performed at dry phase [1,2] by mixing two classes of particles during the fluidisation: target particles to be treated (MP Silica 63–200 Active 6 Å) and ODTCS-carrier particles consisting of coarser porous alumina beads (300–800 μm) containing an adequate amount of reagent.

The aim of this work was to study the effect of the temperature (from 30 to 110 °C) at dry condition on the silanisation reaction rate and the silica surface characteristics. The hydrophobisation treatment is characterized by IR/ATR, Washburn method, elementary analyses and RMN.

The experimental results show that the reaction rate increases with temperature in dry environment but this effect seems to be more significant at lower temperatures. Also, the quantity of reagents can be estimated by measuring the amount of HCl generated by the reaction using a pH sensor. The RMN spectra showed an increased extent of disordering of the grafted chains at lower operating temperatures.

1. Introduction

Silica is an important industrial material used in chemical processing, including chromatographic and catalytic application, and in purification of biologically active compounds. The range of applications for materials made from the silica comes from its surface properties. Silanol groups present on the silica surface cause the surface hydrophylicity to increase. Moreover, the silanol groups allow the surface properties to be changed by introduction of new groups of atoms. These characteristics are important to modify the surfaces by physical or chemical treatment.

One of the most commonly used reagents for silanisation is *n*-Octadecyltrichlorosilane (ODTCS). It is a reagent with three chlorines as functional group and with one long alkyl chain containing 18 carbon atoms, responsible for the hydrophobic behavior. In anhydrous conditions, ODTCS reacts directly with silanol (Si–OH) of silica surface group producing HCl gas, and polymerizes rather horizontally in monolayer pattern. The horizontal grafting corresponds to significant Si–O–Si bridging parallel to silica surface. In humid environment, the polymerization of the reagent can occur before its attachment to the surface [3]. Nevertheless, polymeric aggregates formed by vertical polymerization could attach to the substrate at one or more points through covalent bounds [4]. The information concerning the well ordered monolayer and/or cluster pattern of ODTCS to the solid surface can be obtained through spectroscopic analyses (FTIR, RMN spectroscopy) [5,6].

Despite the large interest of silanisation reactions, their application on industrial scale depends on development of appropriate processes to avoid excessive use of organic solvents. Recent trends in this field aimed at development of alternative solventless methods either in gas phase [1,2] or in vapor phase [7].

The present work deals with the silanisation reaction of highly porous silica powders by a new solventless process performed in a gas–solid reactor. In this process, the chemical grafting is performed in a solid–gas fluidised-bed reactor containing two classes of particles [1,2]. The first class, composed of *target particles,* is the powder to be treated (silica) and the second is composed of the *reagent-carrier* particles with high porosity already containing a known amount of reagent (ODTCS). During the fluidisation, the reagent entrapped within the carrier particles is evaporated and diffuses instantaneously within the fluidised bed. The evaporated

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