DOWNLOAD PDF CHARACTERIZATION OF SOLID MATERIALS AND HETEROGENEOUS CATALYSTS

Chapter 1 : Characterization of Solid Materials and Heterogeneous Catalysts - UCL Discovery

His research field covers physical techniques of catalyst characterization and heterogeneous catalysis for acid- and selective oxidation-type reactions on zeolites and mixed metal oxides. He was President of the European Federation of Catalysis Societies and of the Acid-Base World Organization.

Download TRANSCRIPT 18 Thermal Methods Adrien Mekki-Berrada and Aline Auroux Present-day thermal analysis instruments represent the culmination of a long period of development, including temperature programming and control, controlled fur- nace and sample environment, and data manipulation differentiation, integration, etc. These techniques are applicable to a broad range ofmaterials and inparticular to the study of catalysts, supports and adsorbents. Efforts are made to establish relationship of such techniques with other more accepted or developed methods or to combine them with other analytical techniques in order to handle and evaluate more completely the complex results often provided by thermal analysis. These methods allow us to evaluate the heat involved in thermal transformations, adsorptions or reactions, to measure thermal effects as a function of time, to establish the thermodynamic parameters of unstable states, and to follow the kinetics of changes over a certain range of rates. Further, the redox character of supported metals and metal oxides can also be determined by such techniques, which is illustrated by the case study of DeNOx reactions. Edited by Michel Che and Jacques C. Later developed by Berthelot in , the bomb calorimeter was used for determining the combustion enthalpies at constant volume [1]. Since then, technological evolution has allowed access to new coupled methods and enhanced accuracy. The energies related to the physicochemical changes structural transformation, reaction with the surroundings, etc. A DTA apparatus consists of a pair of crucibles containing the sample and the reference, both encaged in a heating vessel, with control over the atmosphere Figure Crucibles are chosen if possible among good heat conductors which do not react with the sample e. The nature of the thermocouple depends on the temperature range of the analysis e. The recorded signal is in microvolts and is converted to temperature through a quasi-linear law. Usually, calibration is performed with themelting of standardmetallic ormineral substances. Different furnaces and heating elements are available, depending on the tem- perature range: Control of the atmosphere is essential to the accuracy of the experiment. Reactive or inert atmospheres and also vacuum can be used with DTA. In the absence of reaction, the difference in temperature with the heating vessel is low and constant; this signal is called the baseline. Any change related to a heat transfer will be observed as a step glass transition,. Kinetic values can also be derived for an Arrhenius dependence of the rate constant on temperature, and the reaction order model of the conversion function with Kissingers equation [11]: Then the reaction order n is found to be related simply to the square root ofS Figure In addition, two characteristic features are often recorded: However, DTA is more a qualitative than a quantitativemethod. Narayanan and Krishna studied hydrotalcites and hydrotalcite-like compounds as supports to disperse palladium for performing the selectivehydrogenation of phenol to cyclohexanone [12, 13]. Thenature of the support and the method of preparation affect the morphology and the particle size and dispersion of the active metal supported , and therefore the catalytic properties. The differential thermograms of HTx samples show two main transitions which depend qualitatively and quantitatively on the values of x. With an increase in this value, the relative intensity of the lower temperatureendothermicpeakat K, corresponding to the loss of interlayer water, increases and shifts towards higher temperatures for HT0. This indicates that the quantity of interlayerwater and the strengthwithwhich it is bound to the carbonate anions and the hydroxyl sheet increasewith increasing x, that is, the aluminum content of the sample. The high-temperature endothermic peak, which is due to the loss of hydroxyls frombrucite-like sheet and carbonate ion, is shifted towards lower temperatures only for HT0. This could be due to the loosely bound carbonate anion in the interlayer region. This peak is found to occur in two steps inHT0. Since the samples HT0. Nowadays three HT 0. Because of its improved accuracy relative to DTA , and the possibility of having closed containers, it is widely used [14, 15]. A DSC apparatus consists of a pair of crucibles containing

the sample and the reference, both encaged in a heating vessel, with control over the atmosphere. The volume may vary from few microliters to several tens of microliters. For surrounding detector DSC, two thermopiles series of several thermo- couples are shaped in cylinders and installed around each of the crucibles and as much as possible in contact with them Figure The DSC instrument can be calibrated by the Joule effect only for Calvet- type DSC , melting of a standard metal indium, tin, zinc , or heat capacity of sapphire Table According to the tightness and composition of the experimental chamber and of the crucible, all kinds of gaseous environments can be used: Enthalpy changes can be recorded, and also reaction rates. The thermogram can present steps glass transition, etc. In the case of power-compensated DSC, as soon as a deviation from the temperature program is detected, heat is brought to or taken from the sample. This is similar to the concept of an adiabatic calorimeter; the analysis is effected on the compensation signal. The order of reaction n can also be determined via the Kissinger equation see equation For both, either two or three tests can be made: The Continuous Method A sample is heated at a constant rate, while no reaction occurs. The Step Method The temperature program consists of alternate steps of constant heating and isotherm, the latter letting the signal come back to the baseline. Instead of picking a value of evolved heat, it integrates it over the temperature range of the step. The same three steps as in the previous method are observed but with a step temperature program. The performance of DSC can be j 18 Thermal Methods enhanced by coupling with other techniques to obtain better characterization: The decrease in the melting point relative to LiCl was attributed to the formation of eutectic mixtures, and thermodynamic predictions of themetal chloridemixtures gave conclusive results for this assumption Figure It is a very useful technique for any reaction related to a mass loss drying, desorption, reduction, degradation in an active atmosphere,. Even if it is blind to mass conservative phenomena, this can become valuable information when coupled to other techniques, to reveal mass conservative changes, for example a phase transition. A thermogravimetric analyzer consists of a crucible connected to a balance and inserted into a furnace with control over the atmosphere. Crucibles can be made of various materials depending on the temperature range, of different shapes depending on the type of analysis cylindrical is the standard, Figure The quality of the furnace is also of prime necessity. Many aspects are expected: Control of the atmosphere allows numerous possibilities for reactions. Corrosive gases or a vacuum atmosphere will need appropriatematerials and it may then be easier to use a magnetic suspension balance, in order to protect the balance. The key element of the thermogravimeter is the balance. Nowadays it mostly works on the zero method: Three main types can be distinguished: If well adjusted, this can minimize many errors, such as thermal dilatation or aerostatic buoyancy Figure The balance and the container can be separated providing amagnetic suspension setup Figure Many parameters affect the signal: Therefore, themass and scanning rate ought to be kept equal in order to compare results. The derivative of the thermogravimetric signal differential thermogravimetry: Depending on the gas acidic, basic, oxidative,. For example, with a pulse-TG, successive doses of a gas probe can be injected while the effective amount of adsorbed gas is measured by the balance, leading to an adsorption isotherm. The experiments were carried out with a thermogravimetric analyzer. ThePEA thermodesorption curves, collected at different heating rates, were treated numerically in order to interpret them kinetically Kissinger method and derive the kinetic and activation parameters for each type of acidic site of the surface in addition to the number of sites of each type. This allowed the acid sites to be connected to the activation energies for PEA desorption. The peak atTmax,1 can be clearly individuated at any b, whereas the other peak at higher temperature Tmax,2 is better detected at higher b. After purge and heating of the substrate under helium Figure Evidence of the catalytic behavior of iron in the CNT growth process is presented in Figure Themain fact to observe is that the total weight gain in the experiment and weight in each pulse depend on the amount of iron in the sample; the more iron is exposed, the more carbon is deposited. It is also important to consider the different shapes of the thermographs. This suggests that the weight gain due to carbon deposition on the catalyst is a function of the number of exposedmetal atoms acting as catalytic centers and the amount of acetylene pulsed. The simultaneous accuratemeasurement of heat Figure Nowadayssuchdevicesare incommonuseandvarious technological setups

are available on the market Calvet type, plate-DSC,. Although the technology exists, so far themagnetic suspensiondevices aremostly asymmetric one container and only couplings with density or viscosity measurements are provided. Thus mainly two setups can be distinguished: Usual DTA thermocouples, tripod or plate setup can be used Figure Reaction temperature, K, sample weight, 40mg. The weight and the heat or temperature difference are recorded versus time and temperature. Thus the dehydration and degradation temperatures can be obtained, and eventually phase changes corresponding to heat exchanges due to variation of heat capacity, while no mass is lost. Two main mass loss steps were observed: The coupling with FTIR provided valuable information on the online gaseous products during the thermal events not plotted here. The decomposition proceeded generally in two steps, Figure The concepts of heat and temperature are distinct. Heat is only related to temperature by the entropy second law of thermodynamics. Three kinds of calorimetric devices can be distinguished Figure These are devices such as accelerating rate calorimeters ARCs , Berthelot bomb calorimeters and reaction calorimeters in adiabatic mode. Power-compensated DSC is a quasi-adiabaticmethod. The Calvet microcalorimeters, some of the DSCs, and the reaction calorimeters in isothermalmodecorrespondtothiscategory. Reaction calorimeters belong to this category. The apparatus consists of a thermostated jacket in which two symmetrical calori- metric cells are placed sample and reference. Calorimetric cells, or inner vessel, must be inert and good thermal conductors e. Their shapemust adjust to the thermopiles in order to enhance the thermal contact.

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