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Inverse gas chromatography applications: A review

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ABSTRACT

Inverse gas chromatography (IGC) is a versatile, powerful, sensitive and relatively fast technique for characterizing the physicochemical properties of materials. Due to its applicability in determining surface properties of solids in any form such as films, fibres and powders of both crystalline and amorphous structures, IGC became a popular technique for surface characterization, used extensively soon after its development. One of the most appealing features of IGC that led to its popularity among analytical scientists in early years was its similarity in principle to analytical gas chromatography (GC). The main aspect which distinguishes IGC experiments from conventional GC is the role of mobile and stationary phases. Contrary to conventional GC, the material under investigation is placed in the chromatographic column and a known probe vapour is used to provide information on the surface.

In this review, information concerning the history, instrumentation and applications is discussed. Examples of the many experiments developed for IGC method are selected and described. Materials that have been analysed include polymers, pharmaceuticals, minerals, surfactants, and nanomaterials. The properties that can be determined using the IGC technique include enthalpy and entropy of sorption, surface energy (dispersive and specific components), work of co/adhesion, miscibility and solubility parameters, surface heterogeneity, glass transition temperature, and specific surface area.

Contents

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1. Introduction

Inverse gas chromatography (IGC) was introduced in 1941 when Nobel Prize winners Martin and Synge reported using chromatography to measure partition coefficients between two liquids [1]. However, according to Kiselev et al. [2] and Conder and Young [3], the pioneers in applying gas chromatography (GC) to physicochemical measurements were Wicke (1947), Glueckauf (1947), Cremer and Prior (1951), and James and Phillips (1954) who determined adsorption isotherms from GC. The new method got its name in the early 1960s when the term "inverse gas chromatography" was introduced by Professor A. V. Kiselev at the M. V. Lomonosov Moscow State University [4], who played a significant role in developing surface chemistry and chromatographic science [5]. In a book published in 1967 (translated 2 years later) [2], Professor Kiselev and co-authors mentioned the capabilities of GC in determining a number of solid surface properties such as activity coefficients, entropies and heats of solution, vapour pressure, molecular weight, diffusion coefficients, adsorption isotherms, surface free energies, heat and entropies of adsorption, activation energies for internal diffusion and boiling points of hydrocarbons; as well as investigations into molecular interactions and gas–liquid interface resistance. This book and other publications [6–13] indicate that the systematic application of GC in measuring the physicochemical properties of solid surfaces was of great interest during the 1960s. Smidsrød and Guillet named GC as a powerful "indispensable analytical instrument" for applications much more than just determining the components of mixtures [8].

IGC became more popular in the 1970s when it was established as a powerful technique for studying the surface and bulk characteristics of polymers, copolymers and their blends [3,14–27]. IGC in most cases was referred to as a simple, fast and accurate technique for physicochemical measurements, although the term "inverse gas chromatography" was still not commonly used. The number of publications and a wide range of investigations published in the 1980s show that IGC attracted the attention of researchers in a variety of fields [28–31] such as modified silicas [32,33], glass fibres and silicas (as fillers for polymers) [34,35], crackers and sweet biscuits [36]. Polymeric research was still the most common use of IGC based upon the large volume of publications [37–45].

IGC provides information about a wide number of important physicochemical properties such as solubility and thermodynamic interaction parameters; diffusion kinetics; BET surface area; work of cohesion, glass transition temperatures; surface energy heterogeneity; acid-base properties; and polar functionality on the surface of materials as well as characterizing organic adsorbates on particulate surfaces, adsorption isotherms, and work of adhesion. IGC is a valuable method for characterizing the surface properties of the powders dissolving in some solvents, for which inefficiencies with the Washburn technique were indicated, since it determines the interactions between a solid and a liquid without wetting the solid with the liquids [46].

2. Fundamentals

2.1. Instrumentation and methods

Similar to classical GC, an IGC instrument consists of an oven, column, solute reservoir, detector, mass flow controller and a computer as the processor and controller (Fig. 1). Unlike conventional GC columns, which are coils, an IGC column is a straight glass tube; in some research, stainless steel [47–50], copper [48], and teflon [51] columns have also been used. The main difference between the two setups is the nature of stationary and mobile phase. In IGC, the sample of interest is placed into the column, being the stationary phase. The stationary phase may be a crystalline powder, an amorphous compound, a fibrous composition, or viscous liquid. One of the greatest advantages of this method is that no special sample preparation is required. In fact, IGC method necessitates the minimum sample preparation compared to other surface energy analysing techniques [52]. Therefore, various forms of solids and even semi-solids can be characterized quickly and efficiently.

In the case of the stationary phase being a solid form, the technique is referred to as inverse gas–solid chromatography (IGSC), whilst inverse gas–liquid chromatography (IGLC) refers to liquid samples or a liquid stationary phase. According to Davis and Petersen, IGLC was a valuable approach for "fingerprinting" asphalts [6].

To analyse the stationary phase, a low concentration of a well-characterized single gas or vapour of a volatile substance is injected via an inert carrier gas through the stationary phase. This volatile substance is termed the "probe molecule". The direction of gas flow is depicted in Fig. 1. Different probes with different known characteristics such as polarity, acidity, molecular area, and electron donor/acceptor number are used. The respective properties of the stationary phase can be determined by analysing the retention data of the interaction of a well-defined probe with the stationary phase.

The probe is carried through the column by a constant flow of carrier gas which is a high purity inert gas at a set flow rate. The most common carrier gases are helium, argon and nitrogen. In IGC measurements, it is

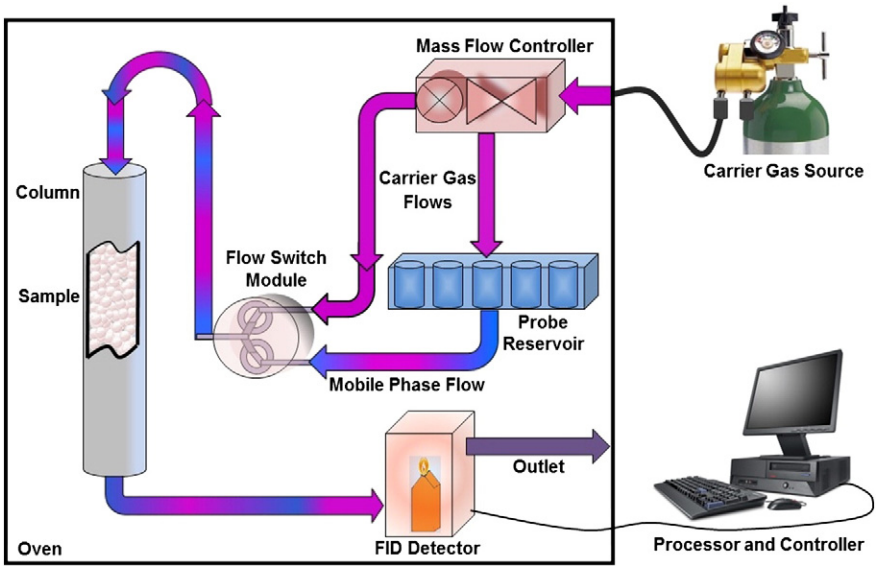

Fig. 1. Schematic illustration of a typical inverse gas chromatography (IGC) analyser.