International Journal for Research in Engineering Application & Management (IJREAM)
ISSN : 2454-9150 Vol-04, Issue-04, July 2018

Analytical Applications of Supercritical Fluids (SCF's)

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Abstract: In recent times, a lot of consideration has been guided towards discovering alternatives to customary solvents. The chemical industry, in general, depends intensely on solvents, which has contributed to the extensive volume of solvent related waste produced to date. This waste is not only harmful to people but can make adverse impacts to the surrounding environment and ecosystems and as such a substitute is required. Some of the proposed thoughts incorporate solid state reactions, greener solvents and alternative solvent systems. An attempt has been made in this paper to focus on supercritical fluids (SCF's) as an alternative solvent system. SCF's are amazingly versatile and have awesome ecological benefits in respect to conventional organic solvents; however the applications of these solvents are not widespread. They are not sufficiently developed to adequately replace solvents used now-a-days. Regardless, SCF's present an area into which certain solvent uses can be diverted in order to mitigate the environmental effects of the current situation while searching for a more practical replacement.

Keywords: Supercritical Fluids, Analytical techniques, Conventional Solvents

I. INTRODUCTION

Nowadays, solvents take up most of the concentration in the field of green chemistry, regardless of whether it is enhancing solid-state reactions, exploring approaches to recycle solvent waste or carry out research on greener solvent systems. Solvents play significant roles in a variety of chemical reactions they are not always essential and at times may obstruct most favorable reaction conditions. Also, the alternate of these conventional systems can reduce the enormous quantity of solvent related waste. For instance, in pharmaceutical industry alone, 80% of the raw materials used in the process are solvents and this is anticipated to increase [1]. Solvents are commonly utilized for their advantages, for example, mass transfer, heat transfer, and dilution of reagents to assure selectivity and potential stabilization of transition states.

The impact of solvent waste of chemical industries on environment emphasizes the need to find out 'greener' and more sustainable alternatives. Supercritical fluids (SCF's) have received a decent measure of consideration in the last two decades. Supercritical fluid alludes to a substance exposed to temperatures and pressures above the corresponding critical point. Carbon dioxide (ScCO2), water (ScH2O), propane (ScC3H8), methanol (ScCH3OH) and nitrous oxide (ScN2O) are the SCFs which are commonly used. ScCO2, is the most commonly used, because of its critical pressure (73 atm) and critical temperature (about 31˚C) [2]. Insignificant environmental impact, non-toxic, non-flammable and relatively cheap is the characteristics which make most SCF an alternative to the conventional systems. An attempt has been made in this paper to review some of the applications of SCF's to evaluate their viability as substitutions to conventional solvents.

II. ANALYTICAL APPLICATIONS

SCF's are mainly used in two analytical techniques namely, Supercritical Fluid Extraction (SFE) and Supercritical Fluid Chromatography (SFC). SCFs offer immense advantages to the previously mentioned techniques because of their alterable solvent properties. The process of liquid extraction can be adapted to accommodate SCF's and is comparatively easy to execute.

Pressure of extraction fluid created by a pump and the fluid is transferred to such a container which remains above the desired critical temperature of the solvent [3, 4]. The relevant analyte is then added to the SCF and the extracted the product, as well as residual SCF are removed. Product and residual are sent to a separate collection vessel which is detained at ambient pressure. Just after transfer of the mixture, the SCF at that point rapidly changes over to its gaseous state leaving only the desired products. The use of SCF's tender manifold advantages such as above the critical point a solvent will exhibit characteristics of both a gas and a liquid [5]. For example, the gaseous nature of SCFs prompts an increase in mass transfer of the solvent. Lower viscosity and larger diffusivity of SCF's are responsible for this enhancement [3]. Because of the decline in mass transfer constraints, extractions carried out with SCF's often required less time to complete the process in comparison to liquid extractions. For example, common SFE's completes in 10–60 minutes while, a conventional liquid extraction can take up to several days [3, 5]. In a

 DOI : 10.18231/2454-9150.2018.0469

study polyaromatic hydrocarbons (PAHs) were extracted from an adsorbent material using both SFE and a traditional extraction method and it was found that both methods offered the similar extraction efficiencies, however SFE demonstrated a reduced extraction time [5].

Also, the solvent properties of SCF's can be modified to a great extent by small changes in temperature and pressure enabling multiple extractions to be performed with one given solvent. This is due to the fact that above the critical point, small changes in the pressure cause significant changes in the density of the solvent [3- 5]. This property is renowned and successive extractions of different compounds using the same SCF have been reported earlier.

Alkanes can be removed at 75 atm in extraction process using ScCO2 at constant temperature whereas up to 300 atm pressure polyaromatic hydrocarbons (PAHs) remain in the sample [6]. At low pressure ScCO2 favors less polar analytes and at higher pressure will favor more polar and higher molecular weight analytes [5]. This shows tunable solvating character of ScCO2. Hence, as contrasting to conventional extractions in which multiple solvents must be utilized to extract all desired products, SCFs offer a convenient technique to eradicate multiple products without changing the solvent but instead altering the physical conditions [3]. Once the analyte has been separated, the SCF is expelled by just bringing the sample to ambient pressure. Conversely, with liquid-liquid extractions, the analyte would then have to be concentrated, resulting in increased energy consumption and man power to get the desired product, which could take place in cases of lengthy concentration steps [3]. In the case of thermally sensitive products, SCFs are the best alternative to conventional solvents as several of them have critical temperatures below 40˚C [3]. Hence, the ability to amend the solvating properties of SCFs with variation in temperature and pressure offers many advantages to SFC. Low critical temperature of ScCO2 allows it for the separation of thermally labile compounds [7, 8]. ScCO2 works best with compounds of low polarity if a small amount of modifier (such as 5–30 % methanol) added to the sample and can be adapted to those of moderately high polarity if required. Also, ScCO2 as the mobile phase is easily applicable to Liquid Chromatography (LC) and Gas Chromatography (GC) instrumentation [8].

In spite of the fact that the applications of SFC may appear to be constrained because of the limited solvating characteristics of ScCO2, it has already in use for the separation of many different compounds such as highly polar antibiotics [7], natural products such as sterols, terpenes, fatty acids and cannabinoids [8] and is even used in chiral separations of enantiomeric compounds [9].
The analytical applications of SCFs are not restricted to the examples given above. It has been shown that SCF's can be used as a part of electrochemical and gravimetric methods [3]. At the same time, the usage of some of these techniques is constrained by the high costs required for somewhat tailored equipment [10]. Though, the examples present the versatile nature of this solvent and underline both the advantages and disadvantages of supplanting conventional solvents with SCFs.

III. Conclusions

Solvents used in most of the chemical industries contribute immensely to the environmental threat. Pharmaceutical industry, automotive industry, textiles, dry cleaning, paints, plastics, rubbers etc. use solvents. In spite of this, it is clear that simply halting the production of solvents would do more harm than good. Therefore, it is essential to discover some alternatives to the conventional methods used. This may be achieved by either make use of greener solvents or removal of solvents from the process overall. The applications presented here evident that there is an opportunity for SCF's to replace conventional solvents. The use of SCF's as the mobile phase in some analytic techniques is a venture to substitute the conventionally used solvents. Though, SCF's cannot substitute all the solvents used these days and solve the present issues. SCF's tenders an eco-friendly changeover state, during which a greener and more viable alternative can be looked for.

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 DOI : 10.18231/2454-9150.2018.0469