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# Extraction of Phytochemicals using Neoteric Solvents

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**Abstract:** *A new era of functional foods has resulted in increased interest in the phytochemicals for preventing and controlling the development of cancer, cardiovascular and other diseases. Besides, the extraction of such chemicals from natural materials by employing processes, which are energy efficient and have minimal impact on environment will be an added advantage. Hence, developing a technically and economically feasible route for extraction of value added compounds from the plant material is the need of the hour. Extractions using neoteric solvents like surfactants, hydrotropes and ionic liquids are such green routes available for a selective, rapid, energy efficient and environment friendly extraction of phytochemicals. The focus of the present review article is to evaluate the influence of such modern extraction techniques for extracting crude fat and phytochemicals from plant matrices utilizing fundamental principles of "green" chemistry. Parameters affecting the extraction processes are also discussed.*

**Keywords:** *Phytochemicals, hydrotrope, surfactant, ionic liquid*

## 1. INTRODUCTION

Natural Products are gaining accelerated importance due to their harmless properties and application in variety of the fields like flavor, fragrance, food, medicine, etc. Furthermore, strict government policy and "green consumerism" have paved path for utilizing more phytochemicals. Each phytochemical is different in nature and has different solubility as well as stability. This is because of different biosynthetic pathways. The synthesis of phytochemicals is governed by numerous factors like environmental conditions, biochemical factors, etc. [1-7]. Depending upon the type of the natural products, different extraction techniques can be employed. Among them, conventional techniques are the established methods for the natural products extraction from the plant material. These techniques include; maceration, percolation, solvent extraction and distillation. Though the conventional techniques are widely accepted, they suffer critical drawbacks as mentioned below.

- Conventional techniques require longer time for complete extraction.
- They consume large volume of organic solvents leading to health and environment concern.
- Residual solvent in the finished product is the major problem in traditional methods.
- It is very difficult to achieve a selective extraction resulting in co-extraction of undesirable compounds.
- There are more chances of thermal or hydrolytic degradation of the sensitive compounds.
- The processes are energy inefficient and affect the environment at a greater extent.

Keeping in mind the issues regarding conventional extraction techniques and the need of green extraction, new techniques should be developed with sustainable approach [8]. The techniques should reduce time of extraction, reduce or eliminate consumption of solvent, reduce energy consumption, increase efficiency and provide automation. Techniques such as supercritical fluid extraction (SFE) [9], pressurized hot water extraction (PHWE) [10], ultrasound assisted extraction (UAE) [11], and microwave assisted extraction (MAE) [12] and hydrotropic extraction (HE) [13-16]. These techniques should provide a sustainable solution by providing reduced time in extraction, eliminating or reducing consumption of solvent, reducing energy consumption, increasing the efficiency and providing automation. A use of green solvents in extraction is very important and surfactant, hydrotropes and ionic liquids fall under this category. These solvents have improved selectivity, easy recovery, easy reusability and environment friendly nature. The extraction can be performed at ambient temperature. Moreover, the solvent extraction technique can be combined with microwave or ultrasound assisted extraction.

This review article discusses the mechanism of extraction using different neoteric solvents and parameters affecting the process. Applications of these techniques are also tabulated.

## 2. EXTRACTION USING SURFACTANT

The surface active agents with long hydrocarbon chain are known as surfactant. When low concentrated surfactant is mixed with liquids, it decreases prominently interfacial/ surface tension or free energy and increases wettability [17].

### 2.1. Principle of Extraction

#### 2.1.1. Extraction from Plant Material

Surfactant has the ability to change the permeability of the cell wall due to reduced surface tension and increased wettability. After which it enters into the cell and dissolve the target compound and comes out upon saturation.

#### 2.1.2. Cloud Point Extraction from Crude Extract of Plant Materials

At distinct temperature turbidity appears in aqueous solution of non ionic surfactant, which is being utilized in this method. Continuous increase in temperature leads to formation of two phases; one is rich phase of surfactant and second is aqueous solution containing surfactant concentration fairly above the CMC (critical micelle concentration). That is known as cloud point temperature (CPT) and this process is known as cloud point extraction (CPE) [18, 19]. In case of natural product extraction using CPE, crude extract is obtained first using organic solvent extraction, which is added in the surfactant solution for CPE.

To enhance the extraction rate as well as efficiency, above extraction methods are coupled with other techniques [20, 21] like microwave assisted extraction, ultrasound assisted extraction or pressurized hot water extraction.

### 2.2. Parameters affecting Solubility

Structure and nature of the solute, addition of electrolyte, temperature, time, concentration, etc. are the factors affecting the extraction process [22]. The parameters which are being studied are discussed below.

#### 2.2.1. Nature of the Surfactant

For selective extraction, a better interaction of surfactant with the target compound is essential. Also, other parameters like viscosity and surface tension of the solution play a major role in facilitating the extraction of the solute. In extraction of piperine using sodium dodecyl sulphate (SDS) and cetyl trimethylammonium bromide (CTAB), SDS gave higher extraction efficiency [23]. In case of extraction of chlorogenic acid (CGA) using SDS, CTAB and Brij-35, a better yield was obtained by SDS [20]. Due to viscosity difference of the two surfactants (Triton X-100 and Genapol X-080), Genapol X-080 solution gave better performance for anthraquinones extraction [21].

#### 2.2.2. Concentration of Surfactant

With an increase in concentration, hydrophobic character of water is enhanced which has effect on the extraction of organic compounds. The variation range of SDS concentration was 0.01–1.0M for extracting CGA, where 0.1M SDS showed highest extraction (2175 mg/100 g of dried mulberry leaves). At lower surfactant concentration, CGA was not properly extracted by smaller number of micelles. With increasing concentration, the extraction efficiency slightly decreases and then remains constant up to 1.0 M SDS. An increase in viscosity with higher concentration might be the reason for decrease in the extraction efficiency [20]. With an increase in concentration of the surfactants, amount of anthraquinones extracted from the roots of *Morinda citrifolia* was found to increase [21].

#### 2.2.3. pH of the Solution

The effect of pH, ranging from 1–9, was studied on extraction of chlorogenic acid using SDS. At pH 1.0, the maximum extraction was observed. The reasons might be attraction between negatively charged surfactant (SDS) and protonated CGA and successive assimilation into micelle [20]. The effect of pH was investigated in the range 1.0–7.0 for extracting anthraquinones from aloe peel powder and the maximal yield was obtained at pH 3.0. Further lowering the pH has resulted in stronger bond between the surfactant and the solute, leading to difficulty in recovering from the surfactant solution [24].

#### 2.2.4. Additives

The addition of electrolyte could increase solubilization of hydrophobic solutes, as a result of this the aggregation number increases [25]. By addition of salt the inaptness between the water structures in hydration and surfactant macromolecules is increased. Meanwhile, it has been well reported that the salt can decrease the cloud point of the surfactant; as a result, the salts enhance the micellar concentration in the surfactant-rich phase. Consequently, extraction yield can be also influenced by the salts. The electrolytes of NaCl, $Na_2SO_4$ and $(NH_4)_2SO_4$ were investigated to enhance recoveries of anthraquinones and their efficacy was found in order of $NaCl_4 > Na_2SO_4 > (NH_4)_2SO_4$ [24]. From black pepper, piperine was extracted, where hydrotropes were used as additives. In case of this a combination of surfactant and hydrotrope gave better percentage extraction compared to the hydrotrope [23].

#### 2.2.5. Temperature

An increase in temperature results in an increase in the extent of solubilization of solutes and reduced viscosity resulting in better mass transfer [22]. Nonionic surfactant solutions may have increasing solubilization capacity due to increase in temperature [25]. At higher temperature, the rupture process of the matrix increases and helps in the process of transfer of the compounds from the cells into the solutions [23]. However, there are chances of extraction of other compounds leading to decrease in purity. In the temperature range of 30 - 90 °C, the percentage extraction of piperine was substantially increased. Within the time span of 70 minutes 98% piperine was extracted at 90 °C. For anthraquinones extraction, some of them are prone to oxidised at a higher temperature, and the extraction yield was affected and, hence, the optimum temperature chosen was 40 °C [24].

### 2.3. Applications

Applications of surfactant are shown in Table 1.

**Table 1**: Applications of Surfactant

| Sr. No. | Plant Name | Target Compound | Surfactant | Highest Yield | Ref. |
|---|---|---|---|---|---|
| 1 | Aloe peel leaves | Aloe anthraquinones | Non-ionic surfactant: Triton X-114 | 93.93% at Temperature: 313 K, pH: 3, Time: 20 min, Alcohol: isobutanol, Concentration: 3%, Additive: NaCl with Concentration: 2% | 24 |
| 2 | Morinda citrifolia roots | Anthraquinones | Triton X-100, Genapol X-080 | 0.97 mg/g material at Surfactant: Triton X-100, Method: MMPHE, Concentration: 1 % (v/v), Temperature: 353 K, Time: 30 min | 21 |
| 3 | Piper Nigrum (Black Pepper) | Piperine | Cetyl trimethylammonium bromide (CTAB) , Sodium dodecyl sulfate (SDS) | 98.00 % at Surfactant: SDS, Temperature: 363 K, Time: 70 min, Concentration: 2 M | 23 |
| 4 | Morus laevigata W. leaves | Chlorogenic acid | SDS, CTAB, Brij 35 | 2.152 mg/g at Surfactant: SDS, Concentration: 0.1 M., pH: 1, Time: 10 s, Microwave power: 50 W, Ratio: 0.2:20 (g:ml) | 20 |

## 3. EXTRACTION USING HYDROTROPES

In the presence of hydrotropes, the process of increasing solubility of organic compounds either water insoluble or scarcely water-soluble in aqueous solutions, is known as hydrotropy. Hydrotropes are made of two functional groups; hydrophilic and a small hydrophobic group.

### 3.1. Principle of Extraction

The mechanism of extraction is similar to that of extraction using surfactant. Penetration depends on the molecular structure of the hydrotrope. The penetration would lead to the interruption or rupture or the cell wall and make it permeable to the solution [13, 14].

### 3.2 Parameters affecting Hydrotropic Extraction

#### 3.2.1. Nature of Hydrotropes

In the process of solubilization of organic substances, the efficiency of a hydrotrope frequently increases with its increasing hydrotropic nature and an increase in extraction can be expected for these hydrotropes. Moreover, the hydrotrope having better interaction with the solute molecule would enhance the solubilization. In case of extraction of *Curcuma longa* from curcuminoids, hydrotropes used are: p-toluene sulfonic acid (PTSA), sodium cumene sulfonate (NaCuS) and sodium n-butyl benzene sulfonate (NaNBBS) [14]. For the dissolution of curcuminoids, NaNBBS was best hydrotrope. But for extraction process it was not suitable because recovery of NaNBBS was very difficult. The reason was very high affinity of curcuminoids towards NaNBBS. It was proved that in extraction of curcuminoids, NaCuS was suitable hydrotrope [14]. Extraction of piperine from *Piper nigrum* was carried out efficiently using NaNBBS compared to sodium xylene sulfonate (NaXS), NaCuS, sodium p-toluene sulfonate (NaPTS) and sodium butyl monoglycol sulfate (NaBMGS) [13]. NaCuS provided better extraction of dioscin from *Dioscorea* rhizomes in comparison with NaXS and NaPTS [26]. For extracting limonoid aglycones from *Citrus aurantium* L. (sour orange), NaSal and NaCuS were used. The maximum limonin yield observed using different hydrotropes were 0.65 mg/g seeds using NaCuS and only 0.46 mg/g seed using NaSal; however, the recovery of aglycones was difficult in case of Na-CuS [27]. The leaves *of Cymbopogon flexuosus* (Steud.) Wats. were used for the extraction of citral. Hydrotropes used for the extraction were sodium salicylate (NaSal) and NaCuS. The maximum yield 15.78 mg/g was obtained using NaSal [28].

#### 3.2.2. Concentration of Hydrotrope

Solubilization capacity of the hydrotrope is higher above the MHC. Higher concentration of hydrotrope leads to decrease in the product purity, increased viscosity of the suspension and paste formation with raw material. The extraction rate depends on the easiness of the hydrotrope to penetrate into the cell wall [13-15].

The extraction of curcuminoids from turmeric increased afar concentration of 0.8 M of NaBMGS. The extraction decreased after its concentration of 1.5 M. The mixing efficiency decreased due to increased viscosity suspension. Due to this poor penetration of hydrotrope has occured [14]. In case of piperine extraction, increase in yield was observed with an increase in concentration but with higher extraction time [13]. Yield was found to be higher as the concentration of hydrotrope was increased for extracting limonoid algycones from sour orange [27].

### 3.2.3. Temperature

Swelling of the cell structure occurs at higher temperature and due to this cell wall become porous to the solution of hydrotrope. The resistance of the cell wall could be reduced. This will have an effect on rate of extraction. A drop in purity at higher temperatures was observed, which is might be due to increased solubilization of other compounds. At higher temperatures, the rate of extraction of curcuminoids into NaNBBS solutions was higher, but at the cost of purity [14]. Similar observations were made in extraction of piperine [13] and limonoid aglycones [27].

### 3.2.4. Particle Size

Size of the plant material is an important factor affecting the extraction. A decrease in the particle size resulted in imporved extraction rate in case of piperine [13] and dioscin extraction [26] but with decreased purity. Though smaller size increases the surface area of the plant material leading to a better mass transfer and thereby better extraction, grinding has an adverse effect on yield. Higher amount of solution was retained by the solid material at lower particle size; hence, a substantial amount of water was used to make the solid cake free of solute.

### 3.2.5. Solid Loading

With an increase in solid amount, the yield was decreased for the extraction of limonoid aglycones from sour orange [27]. The penetration of hydrotrope in matrix was not enough so that it could solubilize the product efficiently.

### 3.3. Applications

Applications of hydrotropes are shown in Table 2.

**Table 2: Applications of Hydrotropes**

| Sr. No. | Plant Name | Target Compound | Hydrotrope | Highest Yield | Ref. |
|---|---|---|---|---|---|
| 1 | Turmeric rhizomes | Curcuminoids | Sodium cumene sulfonate, sodium salicylate, sodium n-butyl benzene sulfonate | 49 % at Hydrotrope: sodium cumene sulfonate, Temperature: 323 K, Concentration: 1 M, 5 % solid loading, Particle size: 6 mesh size | 14 |
| 2 | Orange seeds | Limonin | Sodium salicylate, sodium cumene sulphonate | 0.65 mg/g at Hydrotrope: sodium cumene sulfonate, Temperature: 318 K, Concentration: 2 M, 10 % solid loading | 27 |
| 3 | Lemongrass leaves | Citral | Sodium salicylate, sodium cumene sulphonate | 15.78 mg/g at Hydrotrope: sodium salicylate, Temperature: 303 K, Concentration: 1.75 M, Plant size: 0.25 mm, 5 % solid loading | 28 |
| 4 | Dioscorea rhizomes | Diosgenin | Sodium cumene sulfonate, sodium xylene sulfonate, sodium toluene sulfonate, sodium n-butyl benzene sulfonate, sodium isobutyl benzene sulfonate | 95 % at Hydrotrope: sodium cumene sulfonate, Concentration: 2 kmol/m3, Temperature: 293 K, Particle size: 16 mesh size, Time: 300 min, Solid loading: 5 % (w/v) | 26 |
| 5 | Piper nigrum (Black Pepper) | Piperine | Sodium xylene sulfonate, sodium cumene sulfonate, sodium p-toluene sulfonate, sodium n-butyl benzene sulfonate | 96 % at Hydrotrope: sodium n-butyl benzene sulfonate, Temperature: 300 K, Concentration: 2 mol/dm3, Time: 120 min, Particle size: 710 µm | 13 |
| 6 | Coleus Forskohlii roots | Forskolin | Sodium salts of cumene sulfonate, p-toluene sulfonate, sodium salicylate | 80 % at Hydrotrope: sodium cumene sulfonate, Temperature: 333 K, Concentration: 2M, Particle size: 0.8 to 1 mm | 29 |
| 7 | Embelia ribes | Embelin | Sulfonate hydrotropes: sodium cumene sulfonate, sodium p-toluene sulfonate, sodium xylene sulfonate, sodium n-butyl benzene sulfonate | 95 % at Hydrotrope: sodium n-butyl benzene sulfonate, Temperature: 301 K, Concentration: 3 M, Time: 180 min | 30 |

## 4. EXTRACTION USING IONIC LIQUIDS

An ionic liquid (IL) is a salt in the liquid state. Sometime they are in liquid state near room temperature, known as RTIL (room temperature ionic liquids). They can also be turned as per the application, hence, termed as task specific ionic liquids (TSIL).

### 4.1. Principle of Extraction

In case of extraction using IL, IL selectively dissolves the compound of interest. It has ability to change the permeability of the cell wall and then solubilize the compounds. However complex characteristics of ILs make the extraction mechanism very difficult to explain. In this regard, detailed theoretical background supported by experiments is needed. To enhance the rate of extraction ILs are coupled with microwaves assisted extraction and ultrasound assisted extraction.

### 4.2. Parameters affecting the Extraction

#### 4.2.1. Nature of Ionic Liquids

The physicochemical properties of the ILs greatly depend upon the structure of ILs. This will lead to multiple interactions with the target compound and different dissolution ability for the selected compound. Performance of ILs can be judged by the presence of type of cation and anion in IL and their interaction with the compound. Water miscibility can be determined by the choice of anion. The alkyl chain length can be changed to have better extraction and selectivity of the compound [31, 32].

#### 4.2.2. Concentration of Ionic Liquids

High viscosity of higher concentration leads to poor mass transfer due to inefficient interaction. This was observed when concentration of [Bmim][$BF_4$] was increased from 0.5-1.5 mol/L in extraction of phenolic compounds [32].

#### 4.2.3. pH of the Solution

When pH of [Bmim][$BF_4$] was varied from 1.25-7.00, the extraction efficiency of the phenolic compounds was found to decrease. This might be due to neutralization of pH and reduction in H-bond between compound and IL [32].

### 4.3. Applications

Applications of Ionic liquids are shown in Table 3.

**Table 3. Applications of Ionic Liquids**

| Sr. No. | Source | Target Compounds | Highest Yield | Ref. |
|---|---|---|---|---|
| 1 | Larix gmelini Bark | Proanthocyanidins | 114.86 mg/g using microwave assisted extraction at IL: 1-butyl-3-methylimidazolium bromide-[Bmim]Br, Time: 180 min, Solid-liquid Ratio: 1:20, Power: 230 W, Irradiation time:10 min, Concentration: 1.25 M | 31 |
| 2 | Laminaria japonica | Phenolic compounds | 88.3% using ultrasound assisted extraction at IL: [BMIM][BF4], Concentration: 0.5 mol/L, pH: 1.25, Time: 60 min, Solid-liquid ratio: 1:15 | 32 |
| 3 | Mixed Tocopherol | Tocopherol Homologues | A selectivity of δ-tocopherol to R-tocopherol up to 21.3 was achieved when using [Bmim]Cl as extractant diluted by methanol using LLE at [Bmim]Cl, Mole ratio:1:1.3, Temperature: 303 K | 8 |

## 5. CONCLUSION

Consumer awareness is increasing day by day because of detrimental health and safety aspects of synthetic chemicals and stringent governmental regulations. A use of naturally derived chemicals is gaining importance in sectors as diverse as food and flavors, perfumery, cosmetics and pharmaceuticals. Besides, the extraction of such chemicals from plant materials by employing processes, which are energy efficient and have minimal impact on environment will be an added advantage. Hence, developing a technically and economically feasible route for extraction of value added compounds from the plant material is the need of an hour. Extractions using neoteric solvents like surfactants, hydrotropes and ionic liquids are such green routes available for a selective, rapid, energy efficient and environment friendly extraction of phytochemicals.

In case of extraction using surfactant and hydrotropes, very less focus is given in the area of extraction of natural products, may be because of lack of awareness regarding advantages of surfactants/ hydrotropes or difficulty in selection of surfactants/ hydrotropes. In case of extraction using ionic liquids, they can be adjusted to the targeted compounds because of their properties. Since ionic liquids can be tuned as per the requirement, improved extraction yields can be obtained. Often these neoteric solvents can be recycled, leading to a reduced production cost. Furthermore, extraction using such solvents is not difficult to perform and usually does not require sophisticated equipment. Extraction is often rapid if combined with other novel techniques like microwave and ultrasound assisted extraction.

In conclusion extraction using neoteric solvents may offer the solution to the energy and environment issues with better yield and selectivity bolstered by higher energy efficiency and control on carbon footprint.

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