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Data Article

Extractive separation of protocatechuic acid using natural non-toxic solvents and conventional solvents

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ABSTRACT

Protocatechuic acid is an effective analgesic, anti-inflammatory, antiseptic, etc. The experimental results for recovery of protocatechuic acid using natural non-toxic (groundnut oil and canola oil) and conventional solvents (1-octanol and 2-ethylhexanol) at 300.15 ± 1 K were presented by evaluation of equilibrium, distribution coefficient K_D, extraction efficiency η %, partition coefficient P, and dimerization constant D. Further attempts were made to correlate extraction with physicochemical properties such as dipole moment, dielectric constant, octanol-water partition coefficient, viscosity, density, molecular weight, refractive index, etc. of the solvents. Results were compared with sesame oil, sunflower oil, soybean oil, isobutyl acetate, and 4-methyl-2-pentanone from our previous studies. The K_D and η % values of protocatechuic acid followed the trend as: canola (0.082, 7.41%) > soybean (0.07, 6.56%) > groundnut (0.068, 6.18%) > sesame (0.07, 6.17%) > sunflower (0.05, 4.7%) for natural solvents and 1-octanol (3.392, 75.89%) > 2-ethylhexanol (2.747, 72.88%) > isobutyl acetate (1.46, 58.84%) > 4-methyl-2-pentanone (1.06, 50.51%) for conventional chemical solvents.

Specifications table

Subject area	Chemical engineering, Extractive separation.
Compounds	Protocatechuic acid, sunflower oil, sesame oil, canola oil, groundnut oil, soybean oil, isobutyl acetate, 4-methyl-2-pentanone, 1-octanol, 2-ethylhexanol.
Data category	Spectral and analysis.
Data acquisition format	UV–Visible spectrophotometer
Data type	Experimental and analyzed.
Procedure	The dilute aqueous samples of protocatechuic acid were separated from organic phase after extraction and analyzed in UV–Visible spectrophotometer.
Data accessibility	Data is enlisted in tables within article.

1. Rationale

Protocatechuic acid or 3, 4 – dihydroxybenzoic acid is widely distributed, naturally occurring phenolic acid, found in fruits likegrapes (*Vitis vinifera*) [1], gooseberries (*Ribesuvacrispa* L.) [1], and plums (*Prunus Domestica* L.) [2]. It occurs in

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pigmented onion scales *Allium cepa* [3], carrot (*Daucuscarota*), and mushrooms (*Agaricusbisporus*) [4].The research on protocatechuic acid derivatives (aldehydes, esters, etc.) substantiate its potential of possessing antioxidant [5], antibacterial [6], antidiabetic [7], anticancer [8], antifibrotic [9], and antiviral activities [10] along with exhibiting antihypertensive and cardioprotective effects [11]. Protocatechuic acid also exhibits its utility as a preventive aid in damage by agents causing oxidative stress-mediated nephrotoxicity [12]. The recent findings identified protocatechuic acid to be potent against avian influenza virus H9N2 infection [13] and Infectious Disease Bursal Virus (IDBV) infection [14]. It is a new monomer for synthesizing-bioplastics by recombinant microorganisms from sugars [15]. The electrochemical activity of a composite polymer of aniline and protocatechuic acid acts as an excellent electrode [16].

Protocatechuic acid extraction is difficult from plant secondary metabolite, and to suffice its growing demand, biosynthesis of protocatechuic acid [15–20] is gaining importance. The suggested techniques for its removal from fermentation broth are adsorption [21], microbial degradation [22], ultrafiltration [23], Fenton [24], and H_2O_2/UV or O_3/UV [25]. These methods above have limitations like less selectivity, energy intensive, time-consuming, ineffective for dilute solutions, and generate toxic by-products [26].Immobilization of enzyme onto a solid support for the breakdown of protocatechuic acid in the aqueous stream was found to be effective with no formation of toxic by-products, reduced cost, and time [27]. Extraction is proven and simplest technology which can be employed in normal conditions for the recovery of carboxylic acid.

The recovery of different carboxylic acids by extraction from aqueous phase has been employed effectively such as: gallic acid [28–33], tartaric acid [34], caproic acid [35–38], nicotinic acid [39–40], itaconic acid [41], propionic acid [42–47], benzoic acid [48], phenylacetic acid [49–52], levulinic acid [51], and lactic acid [53–57]. Several research on the use of environmentally friendly organic phase has also been performed such as ionic liquids [58–61], natural diluents [62–63], and novel extractants [64–65].

The current study focuses on protocatechuic acid recovery using natural,non-toxic solvents and conventional solvents to explore its feasibility. Three different categories of solvents such as natural non-toxic (sesame oil [63], sunflower oil [63], soybean oil [63], groundnut oil, and canola oil), dipolar aprotic (isobutyl acetate [71] and 4-methyl-2-pentanone [70]), and polar protic (1-octanol and 2-ethylhexanol) have been used and various extraction parameters were calculated and compared with the physicochemical properties of solvents.

2. Procedure

2.1. Materials and methods

The chemicals used for experimentations are listed in Table 1 and were utilized without any further treatment. Protocatechuic acid (1–10 mmol L^{-1}) aqueous solution was prepared in double distilled water. The range of concentration for protocatechuic acid was accounted for based on the maximum concentration of acid in any aqueous stream and cytotoxicity to human cell lines [27]. The aqueous solution of protocatechuic acid has low initial pH (~3.34) which was advantageous as most unionized protocatechuic acid molecules were recoverable by extraction ($pK_A = 4.48$).

The equilibrium experiments were performed at 300.15 ± 1 K in an orbital incubator (REMI S-24BL, India). An equal volumetric ratio of the aqueous phase (protocatechuic acid) and the organic phase was taken in 100 mL Erlenmeyer flask and shaken for 6 hours to achieve equilibrium followed by centrifugation (REMI 4-RC) at 3000 RPM for effective separation of the phases. The initial and equilibrium pH of the aqueous phase was measured by a digital pH meter (Spectral Lab Instrumental Pvt. Ltd., India). Protocatechuic acid equilibrium concentration was analyzed at 260 nm by UV – VIS spectrophotometer (Shimadzu 1800, Japan). All experiments were performed thrice to check the consistency of results. The experimental procedure of the present work is summarized in block diagram in Fig. 1.

2.2. Experimental uncertainty

The results were reported as mean values and used for further calculations. The replicated results were reliable within ± 2% having an experimental error below ± 5%, and confidence interval of 95% was observed. Standard experimental uncertainty was estimated to be within $x \pm 0.001$ using Eq. (1):

$$\mu(x) = \left(\frac{\sum_{i=1}^{N} \left(x_i - \bar{\bar{x}} \right)^2}{(N-1)} \right)^{1/2} \quad (1)$$

where x_i, $\bar{\bar{x}}$, and N are the experimental values observation, mean of experimental values, and a number of experimental observations respectively.

2.3. Extraction of protocatechuic acid

To extract protocatechuic acid from the aqueous phase, natural non-toxic and conventional solvents were explored. The carboxylic acids in aqueous phase exist as a monomer since the hydrogen bonding of carboxylic acids with water molecules