

Polymeric Membranes and Tea Polyphenols: A Perspective

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Abstract

Tea, derived from the cured leaves of the *Camellia sinensis* plant, is the most popular beverage. It has gained significant attention for its beneficial health properties, including antioxidant, antitumor, anti-inflammatory, and metabolic regulation effects. The main components of tea are tea polyphenols, namely theaflavins and catechins. Tea is categorized into black, green, and oolong tea, with black tea being widely consumed worldwide. Ensuring access to a high-quality life is important, and health and nutritional care play essential roles in human well-being. In tea processing, various clarification methods such as solvent ultrasound-assisted and microwave-assisted extraction are employed. This review examines the features and limitations of these techniques. Membranes have emerged as a prominent factor in this field, offering natural and additive-free ready-to-drink (RTD) tea. The use of membranes for clarifying extracts from black tea, along with their relative advantages and limitations, is discussed. Additionally, this study highlights the connection between tea polyphenols and membranes.

Keywords

Tea-polyphenols; membrane; separation; extraction

1. Introduction

Tea is considered the most extensively consumed beverage in the world. The popularity road map spreads to China, Japan, Europe, India, Sri Lanka, and presently signatures worldwide. The projected black tea consumption will grow at a rate of 3% per annum, reaching 4.14 million tonnes in 2023 [1]. The presence of high antioxidant tea polyphenols and tea flavonoids highlights the health benefits (as antibiotics, anti-diarrheal, anti-ulcer, and anti-inflammatory agents) of this beverage. It can also help treat heart disease, hypertension, vascular fragility, radical scavenging, allergies, and hypercholesterolemia. Perceptions about how human health relates to tea can influence production and tea research. It can be seen as a means to enhance human well-being through enriching diet and nutrition. One of the studies confirmed that drinking oolong tea increases CO₂ exhalation as a substance in the tea operative oxygen inhaling in the mitochondria system [2].

Polyphenols, naturally occurring organic compounds, are derived from the leaves of *Camellia sinensis* [3]. There are

reports of two primary botanical varieties, i.e. Chinese tea shrub (*Camellia sinensis*), and Indian tea tree (*Camellia assamica*) [4,5]. Tea is grown in many countries, mainly China, India, Japan, and Sri Lanka [6]. The most aromatic, top-grade teas are obtained from the undeveloped leaves and young leaflets of the top twigs, collected during the spring season [5]. In 2012, the global tea production reached 4,625 million kg [7]. Tea is primarily classified into three categories: green, oolong, and black [8]. The categories are mainly based on leaf harvesting, leaf maturation, botanical varieties, geographical origin, and agricultural practice. Green tea is processed by drying the leaves, after chopping them, in the sun or with a steam of hot air. In this case, few polyphenols are oxidized as polyphenol oxidases enzyme does not activate properly. Major constituents of green tea include epicatechin (EC), epicatechin-3-gallate (ECG), epigallocatechin (EGC), and epigallocatechin-3-gallate (EGCG), while minor constituents include caffeine, theobromine, theophylline, and phenolic acid (gallic acid) (Figure 1). Oolong tea, on the other hand, undergoes enzyme inactivation through heating after a specific time interval.

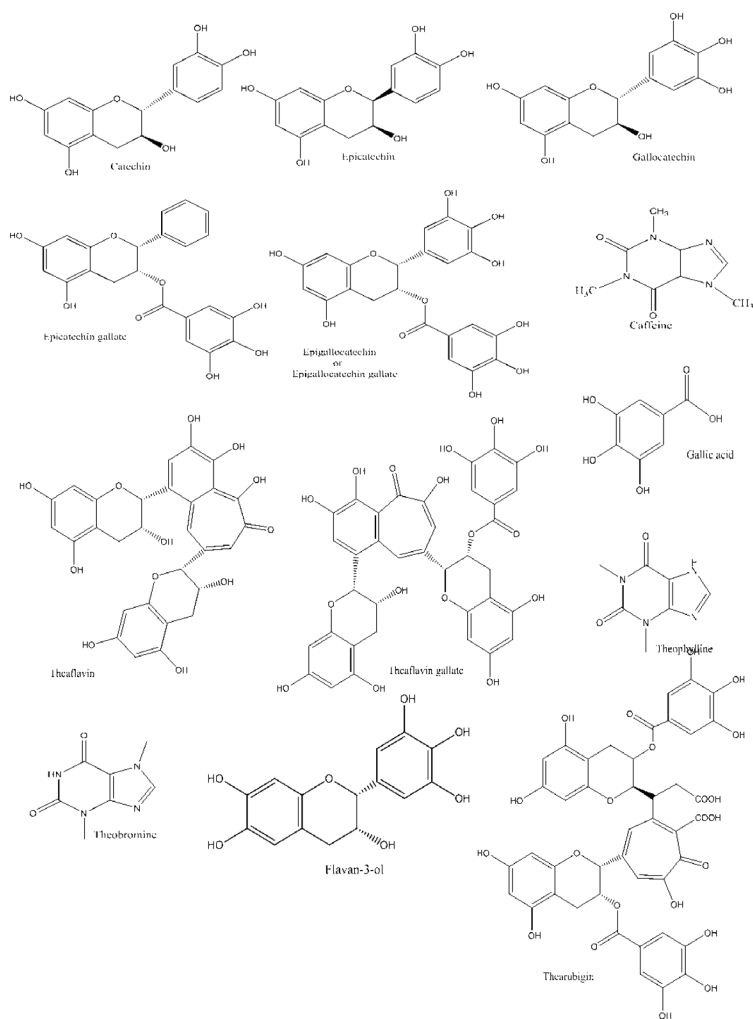


Figure 1: Some chemical structures of tea polyphenol constituents.

Black tea is a fermented product of green tea. The enzyme oxidizes during a period of 3-6 hours after chopping the leaves. The colorless catechins form a complex mixture of polyphenols, mainly the orange-yellow to red-brown i.e. theaflavin-gallate and thearubigins compounds. In this case, 15% of catechins remain unchanged, 10% are transformed into bisflavanols, theaflavins, and other oligomers with a molecular weight of 500–3000 Da, and 75% are transformed into colored theaflavin-gallate and thearubigins compounds having a molecular weight ranging from 700–40000 Da [3]. It is reported that black tea consumption is significantly higher (78%) compared to other tea varieties [9]. Sarkar *et al.* reported that black tea catechin extracts are more effective in scavenging superoxide anions than green tea [10]. Tea production involves various processes such as quantifying polyphenols in natural sources, extraction, and concentration [11–27]. In this context, membrane technology offers a broader range of solutions and effects. As the popularity of Ready-to-drink (RTD) or iced tea gains, the spray-dried tea powder demand increases [28]. The formation of insoluble material termed "tea cream" and haze occurring depends on physical parameters such as temperature, pH, and temperature/time history of the liquor [29]. Thus, removing haze and tea cream-originating compounds from tea is essential. In the fermentation process during black tea production, monomeric flavan-3-ol undergoes an enzymatic

polymerization to a series of colored chemical compounds, viz. theaflavins (TFs) and thearubigins (TRs). These polyphenols tend to interact with proteins and haze. However, the tendency of these polyphenols to interact with proteins leads to haze formation [30]. The decaffeination methods are based on the removal of the precipitating complexes. It includes clarification by centrifugation/filtration and a combination of the methods. One of the approaches increases solubility by adding chemicals and enzymes [31].

Membrane processes could be a better alternative for the separation and concentrating process, with potential advantages such as better separation efficiency, high purity, mild operation parameters, temperature independence, and reduced or avoided use of solvents. There are various options such as ultrafiltration and nanofiltration are available [3]. Moreover, it can be flexible to result in hybrid techniques. The separation science and membrane technology coming together is a moment bristling with possibilities and challenges.

2. Determination of Polyphenol Content

The well-acquainted few methods with their features and limitations for the determination of polyphenol content are summarized in Table 1 [32].

Table 1: Different analytical methods for determining polyphenol content.

Analytical Methods	Features	Limitations
Titration method (oxidation by permanganate) [33]	Simple process and easy to do anywhere	Other compounds (viz. non-polyphenols) may participate in the oxidizing reaction Endpoint detection
Spectrophotometry [34–36] a) Ferrous tartrate method [37] b) Folin–Ciocalteu method [38]	Widely used, the right selection of the chromogenic agent, and good accuracy	Different polyphenols show differences in color formation ability (especially Fe(III) compounds) which makes it difficult to monitor the right conversion coefficient for the measurement It needs extensive pretreatments
Infrared spectroscopy	Non-destructive and no need for chemical reagents. Rapid detection abilities	It needs reliable calibration
Electrochemical analysis [39]	High precision and sensitivity, quick analysis, make-in miniature scale, and on-site detection	It needs electrodes of high precision [40]
Liquid Chromatography [41]	Good separation performance and better sensitivity	Simultaneous determination of individual polyphenols and separation of individual tea phenols is difficult from the complex systems [42,43]
Liquid Chromatography with Mass spectrometry (LC-Mass) [44,45]	Sensitivity, precision, and better resolution [46]	It needs the expertise to analyze the spectra

2.1. Potentiality of Black Tea

Tea components possess antioxidant, antimutagenic, and anticarcinogenic effects. Black tea catechins provide some protection against degenerative diseases. They have antiproliferative activity on hepatoma cells (i.e., cancer-liver cells) and hypolipidemic activity in hepatoma-treated rats. Catechins could prevent hepatotoxicity and mammary cancer post-initiation [47]. Consuming black tea can counter many types of cancer, including lung, colon, esophagus, mouth, stomach, small intestine, kidney, pancreas, and mammary glands [48]. Meydani *et al.* reported that black tea could protect against cancer from environmental agents [49]. Several studies and clinical trials showed that black and green tea (Oolong tea to a lesser extent) could reduce the risk of many chronic diseases [50]. Black tea may lower blood pressure and thus reduce coronary heart disease risk. An interesting observation based on animal studies is that it reduces blood glucose levels and body weight, thus protecting against coronary heart disease [51]. Mukhtar *et al.* reported the inhibitory effects of black tea leaves against tert-butyl hydroperoxide-induced lipid peroxidation [52]. A similar antioxidant impact on the kidney is observed after oral administration of the significant tea polyphenol EGCG. The formation of peroxides is reduced more effectively by crude catechins compared to dl- α -tocopherol [53]. The chemopreventive effect of black tea among cigarette smokers is also reported [54].

Black tea is associated with increased bone mineral density and protects against hip fractures [55]. It strengthens the immune system's action by protecting it from oxidants and radicals. It is helpful for insect stings due to its anti-inflammatory effects and capacity to stop bleeding [56,57]. It reduces the risk of kidney stone formation [58]. The catechins in black tea have an inhibitory effect on *Helicobacter pylori* infection [59]. Black tea can counter the influenza virus, especially in its earliest

stage, and the *Herpes simplex virus* [60–62]. Mukoyama *et al.* observed the *in vitro* inhibition of adenovirus infection by black tea catechins [63].

Green tea catechins show their behavior as antitumorogenic agents and immune modulators in transplanted tumors of carcinogen treatment [64]. Green tea extracts effectively prevent oxidative stress and neurological problems [65,66]. Green tea can counter diarrhea and typhoid [67–69]. Hirasawa *et al.* observed the positive effects of green tea extracts and green tea polyphenols (GTPs) on the proliferation and activity of bone cells [70]. The proliferation of hepatic stellate cells is closely related to the progression of liver fibrosis in chronic liver diseases. EGCG has a potential inhibitory effect on the proliferation of these cells [71,72]. Recent studies suggested that GTPs might counter Parkinson's and Alzheimer's diseases, and other neurodegenerative diseases [73,74]. Gupta *et al.* reported that green tea preserved the lens antioxidant defense system in an experimental cataractogenesis model [75]. It shows a beneficial effect of green tea on alcohol intoxication [76].

2.2. Extraction of Tea Polyphenol

Tea polyphenol extraction is the art of restoring volatile compounds, avoiding biodegradation, and maximizing recovery [77–79]. In this area, a few methods are explored including solvent extraction, microwave-assisted extraction, ultrasound-assisted extraction, high-pressure processing, supercritical fluid extraction, and subcritical extraction [80]. Some of the methods are summarized below.

2.2.1. Solvent Extraction

This method is based on the relative solubilities of the compounds in a particular solvent. Phytochemicals are recovered from tea polyphenol using different solvents (e.g. water, alcohol, acetone, and dimethyl formamide) [81].

Mostly Soxhlet, maceration (softening by soaking in a liquid), reflux, and hydrodistillation techniques are employed [82].

Features

- Simple method
- Not location specific

Limitations

- Chances of thermal degradation of active components
- Using organic solvents is not environmentally friendly [83]

2.2.2. Ultrasound-Assisted Extraction

The tea polyphenol solution mainly propagates ultrasound pressure waves (high frequency ~20 kHz). It forms bubbles that generate microturbulence, disrupt cell membranes, and promote solvent dissolution [84]. Different influencing factors are extraction efficiency, time, solvent composition, and input power [85,86].

Features

- Low-temperature extraction
- Time-saving
- Minimizes solvent use
- Can be coupled with other extraction techniques

Limitations

- Needs sophisticated arrangement
- Energy consumption relates to process efficiency
- Restricted to a definite volume

2.2.3. Microwave-Assisted Extraction

Microwaves form energy by ionic conduction and dipole rotation, resulting in internal water vaporization that disrupts the biomass cell wall and plasma membrane. It uses microwave energy to heat solvents and partition components from the natural sample matrix into the solvent accordingly. The permeability of solvents to the cell matrix is enhanced, allowing the dissolution of molecules. The efficiency of the process depends on the polarity and volume of extracting solvent, volume, temperature, time, and microwave power [87]. Li *et al.* experimented with different parameters in this aspect and reported the irradiation time > intensity > tea/water ratio > number of times for irradiation [88].

Features

- Clean process
- Efficiency relates to the optimization of parameters
- Applicable to thermally unstable substances

Limitations

- Restricted to organic solvents
- Energy consumption relates to the efficiency of the process
- Restricted to a definite volume
- Risk of bio-component degradation

2.2.4. High-Pressure Processing

This method is based on adiabatic heating, which increases solvent permeability and extracts solubility. It is used for the

extraction of tea polyphenol from plant materials at high pressures of 100–800 MPa or <1000 MPa. It is a fast and effective method [89].

Features

- Non-thermal process [90]
- The time of extraction can be minimized
- High extraction efficacy
- Variable options like ultrahigh and high hydrostatic pressure [91]
- Energy consumption is low in mild extraction conditions [92]

Limitations

- Possibility of structural changes of the bio-components [93]
- Potential undesirable sensory changes in foods [94]

2.2.5. Supercritical Fluid Extraction

This method separates extracts from the matrix using supercritical fluids as the extracting solvent. It is based on supercritical fluid (viz. CO₂). It results in lower viscosity and higher diffusivity above its critical temperature. A packed bed adsorption column with the help of fluidized CO₂ by high pressure is used for the extraction.

Features

- Green solvent
- Time consumption can be minimized
- Heating and evaporation can be avoided [95,96]
- Avoidance of toxic organic solvents [97]
- The supercritical fluid (CO₂) has a lower critical point, abundant in nature, and can be recycled
- Can use co-solvent and modifiers to enhance extraction yield [97]

Limitations

- Unavoidable loss of tea catechins [98]
- Possibilities of structural changes of the components

2.2.6. Membrane

Membrane clarification of green tea extract is a promising step to improving its quality. Membranes of different ranges (micro to reverse osmosis) are used in the tea polyphenol arena. Membranes have the ability to separate particles into organics depending on their pore size. Micro and ultrafiltration separate particles and large macromolecules whereas nanofiltration and reverse osmosis separate organics of different sizes. Tea polyphenol is composed of different organics, as discussed earlier. There is the possibility of formation of complexes as well as oligomers. The features and limitations of different membrane processes in this arena are in the ensemble (Table 2).

Table 2: Different membrane processes with their features for tea polyphenol separation.

S.N.	Membranes	Pore size range (nm)	Features
1	Microfiltration (MF)	100 - 1000	It has the ability to separate suspended solids or colloidal particles, bacteria, oligomers, thearubigins, and turbidity. It needs low operating pressure and thus requires low energy.
2	Ultrafiltration (UF)	10 - 100	Suspended solids and solutes of high molecular weight (viz. caffeine, gallic acid, epigallocatechin gallate (EGCG), polyphenols, carbohydrates, catechin in oxidized form, polysaccharides together with oxidized polyphenols, polymerized intermediates, and other complexes, proteins are separated in clarification [27,99].
3	Nanofiltration (NF)	1 - 10	It has the ability to separate organic compounds (viz. pectins, proteins, chlorophylls, thearubigins, theaflavins, and other oxidative products that form complexes with residual metal ions, catechins in oxidized form, amino acids) along with higher valent salts effectively [99].
4	Reverse Osmosis (RO)	0.1- 1	Loose Reverse Osmosis membranes are capable of separating organics (in their non-oxidized form) along with salts.

Membrane technology can overcome some disadvantages inherently associated with the conventional decaffeination methods, i.e., removal of precipitating complexes. Membrane separation processes are better alternatives for concentration and separation processes with potential advantages of greater separation efficiency, high purity bioactive phenolic compounds from the respective streams due to their flexibility and mild operating conditions, and avoidance or reduced use of solvent. Different polymeric materials (viz. cellulose, polysulfone, polyamide thin film composite membrane) are used for membrane preparation.

Chandini *et al.* experimented broad range of MF (200 and 450 nm pore size) and UF (25, 50, 100, and 500 kDa) membranes [100]. The rejection of tea components increased with the decrease in pore size. The clarity of membrane-processed extracts was excellent 4 NTU (Nephelometric Turbidity Units). UF-500 and MF membranes showed better retention and more significant recovery of tea solids, including polyphenols in the clarified extract. The rejection (%) of catechin and caffeine with different UF membranes (viz. Polysulfone, flat sheet (GR51pp, DDS (Dow Danmark A/S), polyether sulfone, flat sheet PTHK, Nihon Millipore, Ltd., PTTK, spiral, Nihon Millipore, Ltd.) is promising though they are small molecules [101]. The pore size range is 0.014 to 0.008 μm . The separation (viz. $R(\%)_{\text{Catechin}}$ 3.6 to 10.8) increases with the decrease in the pore size of the membranes.

Evans *et al.* experimented with the performance of fluoropolymer (from Alfa Laval Nakskov) and regenerated cellulose (from Microdyn-Nadir) membranes having MWCO 30KDa [101]. Two membranes could remove 21% and 27% tea solids having flux 23 and 32.1 LMH respectively at 1.0 bar. The permeate lightness and yellowness increased, and thus haze decreased. The concentration polarization resistance was significantly higher for regenerated cellulose membrane compared to fluoropolymer membrane.

The clarification performance of black tea depends on the physical features (hydrophobicity, surface charge, and surface roughness) and the chemical properties of the membranes [102]. In this regard, they have investigated regenerated cellulose (RC) and fluoropolymer (FP) membranes supplied by Microdyn-Nadir. The fouling increased on the rougher, more hydrophobic FP membrane surfaces. It showed the order FP30 (59nm) > FP10 (27nm) > FP100 (11nm). The foulants were significantly entrapped on the rougher surface of the membrane. It also correlated with the negative charge. FP membranes were rougher compared to RC membranes. Thus, less deposition occurred on the RC membranes. The membranes with the isoelectric points at the pH of the liquor showed a higher fouling tendency. The fouling mechanism resulted in cake deposition on the membranes. The total fouling resistance depended on the ionic strength and increased concentration polarization. Adding calcium to the black tea feed solution resulted in polar group-calcium complexation (bridging). It increased irreversible fouling [103].

Cross-linked polyvinyl alcohol was coated on asymmetric polysulfone (PS) support (MWCO 6 kDa), showing the potential for separating tea polyphenol [104]. The cross-linking between poly(vinyl alcohol, PVA) and dibasic acid (maleic acid, MA) was done for the coating. Figure 2 depicts the schematic diagram of the coated membrane. The membranes showed 70-90% polyphenol retention having a flux 15-20 LMH at 500 psi for various coating and support types. The modified membrane develops the reduction in fouling, and at the same time, the pore size decreases compared to the support membrane, without much compromising productivity flux. It showed steady performance in water or methanol at pressurized conditions. The variation of flux with time showed that all the membranes are steady with no loss in flux during a methanol exposure time of 250h.

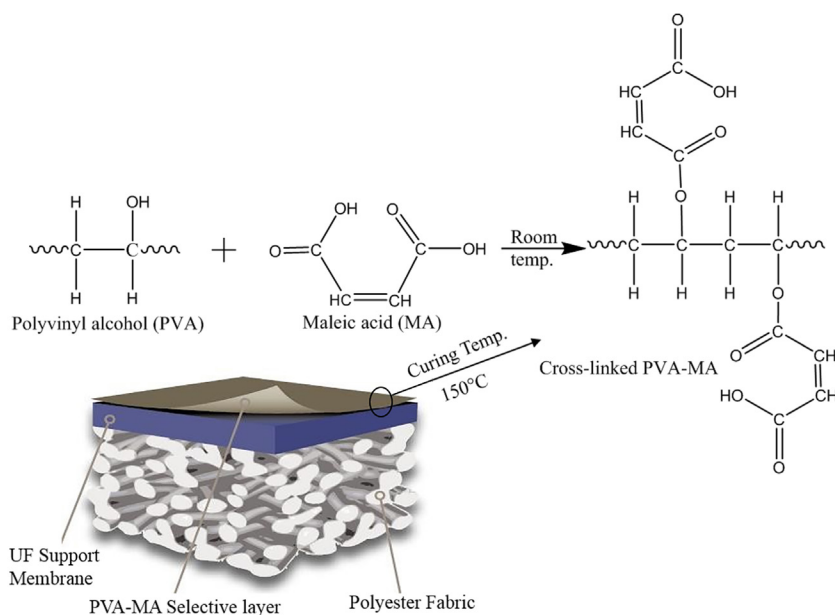


Figure 2: Schematic diagram of the coated membrane (PVA-MA).

The efficiency of the hollow fiber microfiltration membrane to enrich the epigallocatechin gallate (EGCG) from green tea aqueous extract was reported [105]. The particle size distribution of the raw and hollow fiber microfiltered (av pore diameter 0.1 μ m) green tea extract is observed that the range of particles in the extract was 40-100nm suggesting that the membrane passes the smaller-sized solutes (≤ 100 nm) [105].

The flux decline of membranes was quantified with simple resistance in the series model. The study showed that the growth of the fouling layer with the time of filtration is fitted with the first-order kinetic model.

$$R_f = R_f^s [1 - \exp(-kt)]$$

Where k designates the proportionality constant and represents the growth rate of the fouling layer. Larger ' k ' features a rapid increase of RF to the steady state, and small ' k ' signifies slow growth of fouling resistance.

The fouling resistance of the membrane improves with transmembrane pressure (TMP) drop and declines with Reynolds number.

Wu *et al.* studied the filtration characteristics of model solutions containing tea proteins, theaflavins (TFs), thearubigins, and caffeine through PS UF membranes (MWCO 30kD, GE Osmonics, Minnetonka, MN) [106]. The permeate flux declines rapidly i.e. > 60% of the initial flux was lost within 15 min. Pure TF filtration creates concentration polarization. The mixture of protein or protein mixed with polyphenol with caffeine reduces normalized filtration fluxes significantly.

The adsorptive fouling of polyphenolic compounds on UF membranes was also seen [107]. It mainly depends on membrane-solute interactions and membrane-solute-solute interactions. It might be through H-bonding, hydrophobic interactions, benzene ring interaction by p-p stacking, and changes in water structure at the membrane polymer

surface. The nature of membranes (chemistry on the surface and pore size) and the nature of feed (pH and salt content) control the adsorptive fouling. Due to the fouling, the water flux reduction takes place. The impact of water flux reduced due to polyphenolic compound (PPC) adsorptive fouling was higher than protein fouling. Ethanol treatment of polymeric membranes to improve the performance of multiple foul-clean cycles is also explored [108]. It facilitates better transmission of polyphenolic compounds and surface charge of the membranes, though reducing the negative surface charge following treatment resulted in detrimental performance. The performance of the nanofiltration membrane (NF 270 (MWCO 270) and NTR-7450 (MWCO 700)) was analyzed. NF 270 membrane showed 90% rejection, whereas NTR 7450 membrane experienced 94% rejection [109].

The combination of ultrafiltration and reverse osmosis treatment for the clarification and concentration of tea extracts is explored [99]. The catechin content was high in the retentate >100KPa pressures in all types of filtration processes studied compared to the original content of fresh green leaves. Rao *et al.* developed a hybrid technique consisting of ultrafiltration, silica gel, and chitosan treatment for clarification [110]. The silica gel and chitosan treatment decreased haze-active protein significantly. The post-treatment through ultrafiltration (10 kDa regenerated cellulose membrane, Shanghai Mosu Instrument Co., Ltd. (Shanghai, China)) reduced the protein content considerably from 11.36 to 5.85 mg/100 mL. The regenerated cellulose commercial membranes were used.

3. Future Directions

The convergence of tea polyphenols and membranes presents a pivotal moment brimming with possibilities and challenges in the health sector. These two factors stand as the primary catalysts for rapid advancements in this field. The continuous improvement of membrane technology to enhance performance is an ongoing endeavor. One of the key areas of focus is the mitigation of fouling on the membrane. Researchers have been diligently orchestrating the overall

direction, particularly in terms of hybrid technologies. However, a systematic approach is required to fully exploit this potential.

4. Conclusions

Perceptions regarding the relationship between human health and natural resources can significantly influence the direction of research. Leveraging natural resources has the potential to promote well-being and prevent diseases. By combining these resources with advanced technologies, greater benefits can be achieved for individuals. In this context, the quest for improved methods of tea clarification has been discussed, including their unique features and limitations. Membrane techniques have emerged as a prominent solution, as they enable the production of natural and additive-free ready-to-drink (RTD) tea. Additionally, specific membrane types such as ultrafiltration and nanofiltration, along with their respective performance capabilities, have been highlighted.

Authors' Contributions

Mayank Saxena: Literature survey and writing. A. Bhattacharya: conceptualization, drafting, editing, and communication.

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Conflict of Interest

The authors declare no conflicts of interest.

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