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Recent Advances in Techniques for Flavor Recovery in Liquid Food Processing

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Abstract Recovery of volatile flavor-active aroma compounds which are key components of processed liquid food streams is of utmost concern to food industry, as these compounds contribute to the quality of the final product. This review paper highlights the recently published research on different techniques that can be applied for recovery of the key flavor components which all aim for minimizing the loss of volatile aromas and (re-) using them in process streams, in order to enhance the flavor profile of the liquid food product. Among the available techniques for flavor recovery in food industry, distillation or stripping, pervaporation, supercritical fluid extraction, and adsorption showed potential for selective recovery of the flavor components from liquid food streams. These techniques can be combined in different stages of the process or applied as an alternative to the other techniques for aroma recovery. Less attention has been paid to supercritical fluid extraction among the available techniques, especially for recovery of aroma components from alcoholic beverages. Since this technology demonstrated high selectivity for flavor recovery in fruit juices and can take profit from applying natural solvents like CO₂, further research on the application of this technology combined with counter-current flow in a multi-stage contactor is recommended to optimize the recovery process. Adsorption also shows potential for flavor recovery that can be combined with thermal processing or applied as an alternative stand-alone technique.

Keywords Volatile flavor components · Selective recovery · Liquid food streams · Alternative techniques

Introduction

Flavor perception is the sensory impression of food or any other chemical substance, determined by chemical senses of taste and smell [81]. Flavors are a mixture of volatile aroma compounds which are classified to natural, natural identical, and artificial flavorings, [13, 37, 53, 96]. Different chemical substances contribute to particular flavor perceptions [37, 55] as is depicted in Fig. 1. Flavor-active compounds, which are normally present in beverages and liquid foods, are various organic compounds, typically present at low concentrations (ppm levels). Different classes of these organic compounds, which can be regarded as aromas, for instance, are *aldehydes* [36, 55, 95], *esters* [23, 57, 106], *carboxylic acids* [27, 98], *phenols* [26, 49, 91, 110], *hydrocarbons* [76], *ketones* [34], and *terpenes* [31, 54]. These flavor-active components are widely used in beverage industry with the largest market in North America, followed by Asia-pacific and Europe [59]. These markets are highly mature and emerging in Latin America and Eastern countries. Approximately a growth rate of 5% is projected to food flavor market since 2015 and continuous growth is expected till 2020 [60].

The value of food flavor market is projected to reach USD 15.1 billion by 2020 [6, 60]. Several alcoholic drinks such as wine, beer, cider, and spirits are available in the market, in which global top players account for share above 60%. Flavored alcoholic beverages (FABs) share an important market segment based on different age groups and beer, cider, and FABs dominated the global market in 2014 [10]. Considering the growing demand for flavor-active components' consumption in food and beverage industry, it is of extreme importance

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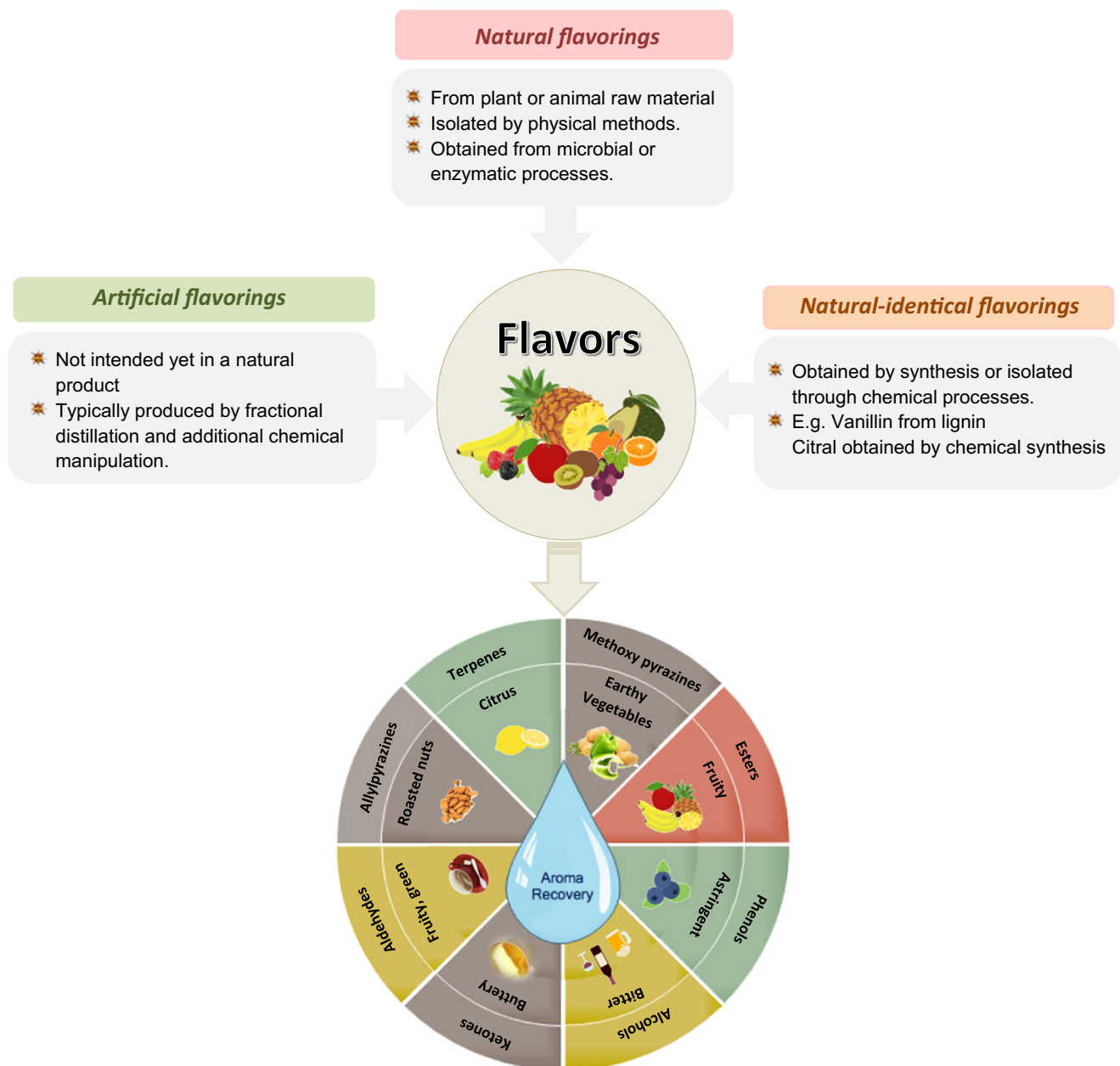


Fig. 1 Classifications of flavors in food and beverages and contribution of chemical compounds to various flavors [13, 40]

to quantify and control the level of these compounds accurately. The main flavor-active compounds present in liquid foods and beverages together with their physical properties (hydrophobicity and solubility) are presented in Fig. 2. The functional groups (groups responsible for chemical reactions) for each molecule are illustrated in red color. The higher value of partition coefficient indicates more hydrophobicity of the flavor compound and less solubility in water can be achieved [75].

During processing, the flavor composition of the beverage might alter to a great extent, due to chemical and physical changes of the aroma complex [52]. Chemical changes might occur due to oxidations or Maillard reactions [67], during heat

treatment that can result in losses of the flavor compounds or formation of new flavor compounds from original flavors. Physical changes in the flavor composition can also occur during concentration and removal of the excess water, while some amounts of the volatile flavor compounds like esters might be lost due to evaporation. These changes in flavor composition are considered as undesired, and in order to prevent or reduce the unwanted changes in composition of flavors, different techniques can be implemented, which take advantage of the physical properties of flavor-active components like solubility, relative volatility, and hydrophobicity for their separation (as explained in Fig. 3). To reduce the

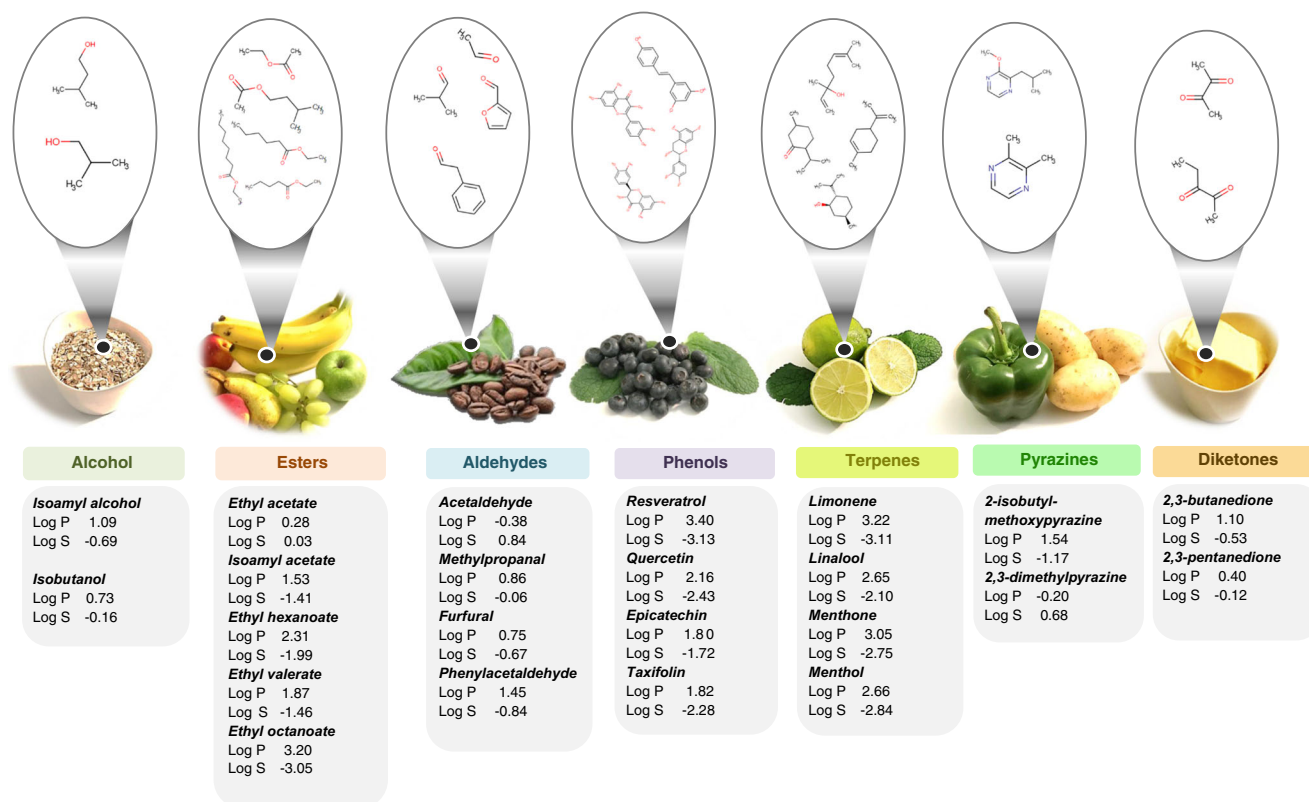


Fig. 2 Main flavor-active components in liquid foods and their physical properties (Hydrophobicity and solubility) (ChemAxon) [20]. The Partition coefficient (P) is the ratio of concentrations of an un-ionized compound in the two phases of immiscible solvents (water and

n-octanol) at equilibrium. logP is the 10-base logarithmic measure of the partition coefficient; log S or intrinsic solubility is the 10-base logarithmic measure of the solubility (mol/L)

unwanted changes and losses of flavors during processing, volatile aroma compounds, found in different side-streams of the process, can be selectively recovered or removed from the raw material prior to processing, or improvements in the design can be implemented to achieve the desired recovery. In the latter approach, achieving the desired process option is not always possible and many factors should be taken into account in order to design the appropriate process which is also feasible in terms of costs in comparison to traditional process. Alternative techniques can be applied and have been proposed by researchers to enhance the aroma recovery, which all aim for minimizing the aroma loss, by producing an aroma concentrate which can be put back to the final product and consequently improves its sensory quality. This paper serves as a summary, with the aim of giving an overview of the research and developments in techniques that are being applied for aroma recovery in liquid food process industry in recent years.

Techniques for Flavor Recovery

Recovery of the volatile aroma components is practiced in processing of fruit juices, alcoholic beverages, and other

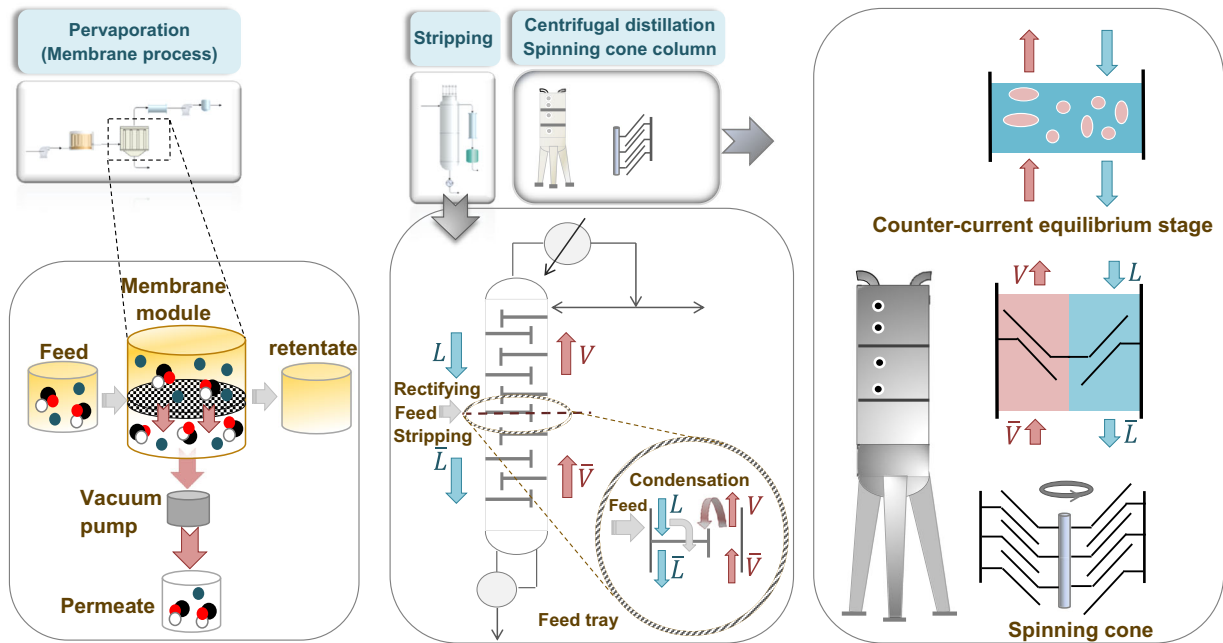
liquid food streams and is usually connected with evaporation [79, 101, 108]. It is mainly performed by stripping or distillation processes (based on differences in components' relative volatility) and also other alternatives like pervaporation (using vapor and liquid phase and a membrane), supercritical fluid extraction (using liquid/ solid and a supercritical fluid), and adsorption (using solid as auxiliary phase and liquid). Fig. 3 represents the available technologies for aroma recovery in liquid food processing. An overview of current research advances in each technology is provided in the next sections.

Aroma Recovery Through Distillation

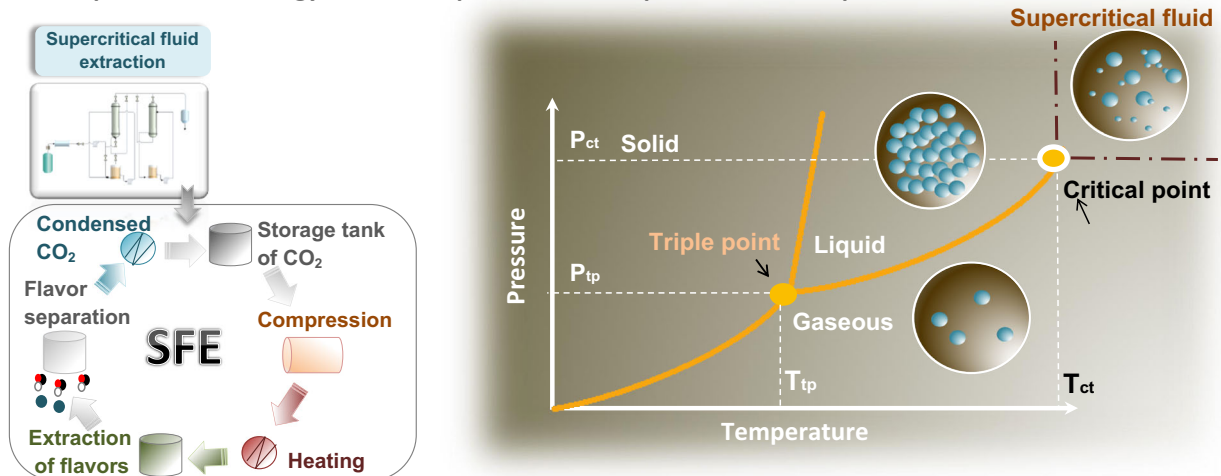
The principle of the classical distillation system is stripping the aqueous food stream containing the most volatile compounds and concentrate them by fractional distillation to a solution about 100–200 times the original concentration [86]. It usually combines stripping with rectifying and enrichment of the volatile aroma compounds [61] (represented in Fig. 3 (part a)), in which the former depends on the relative volatility of the aroma components. An impressive number of research works contribute to our understanding of the application of this technology for flavor recovery from liquid food

Alternative technologies for flavor recovery

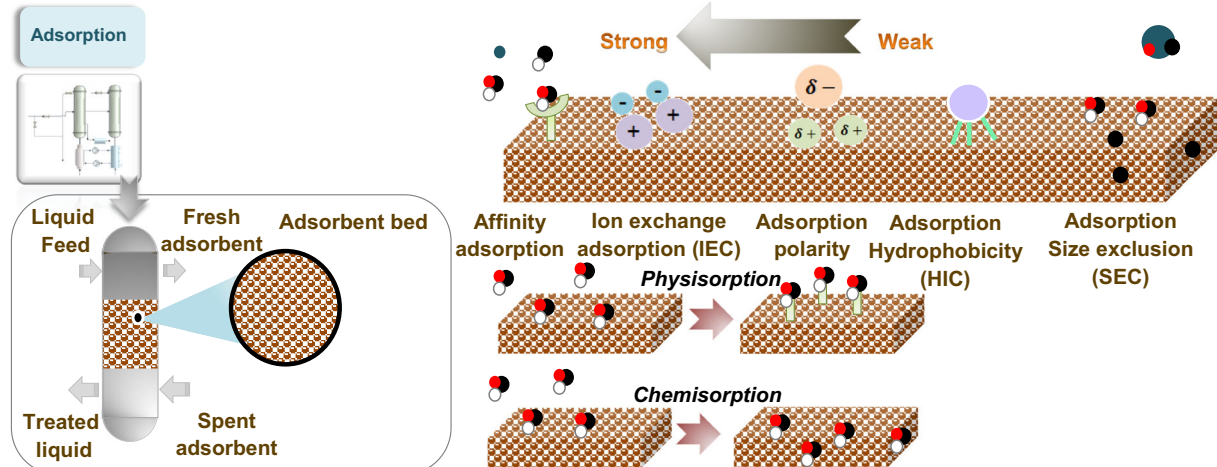
a Separation technology based on vapor/liquid equilibrium



b Separation technology based on liquid/solid and supercritical fluid equilibrium



c Separation technology based on solid/liquid equilibrium



◀ **Fig. 3** Alternative technologies for flavor recovery: **a** vapor/liquid equilibrium (pervaporation, stripping, and centrifugal distillation), **b** liquid/solid and supercritical fluid equilibrium (supercritical fluid extraction), and **c** solid/liquid equilibrium (adsorption) [24, 71, 88]

and beverages [4, 5, 21, 33, 35, 38, 45, 63, 66, 68, 89, 93, 94, 99]. Few recent research works unequivocally demonstrated the application of Membrane Distillation (MD) and Vacuum Membrane Distillation (VMD) for flavor recovery during the last 4 years [1, 21, 68, 77]. Performance of MD is investigated during beer dealcoholization processing and the effect of feed and vacuum pressure are investigated on flux and selectivity of a thin-film composite polyamide membrane. The increase of feed and vacuum pressure could improve the membrane flux, but decreased the membrane selectivity [77] and no major change in composition of the flavor components, maltose, and glycerol was observed, only slight loss of maltose in dealcoholized beer was related to the adsorption phenomena on the membrane surface for which membrane flushing for recovery of the flavor compound was proposed. In comparison to MD configurations, VMD is believed to be an attractive and cost-competitive technology, besides being characterized by a lower operating temperature and hydrostatic pressure. It permits higher partial pressure gradients; therefore, higher permeate flux can be achieved [1, 35, 99].

The application of this technology is investigated recently for fractionation and separation of hydrocarbon terpenes of green mandarin from alcohols, ketones, and aldehydes [93].

The influence of column pressure on boiling point of essential oil and the composition of compounds in distillate is studied. According to this study, efficient separation of terpenes could not be achieved unless higher number of stages are used and no major degradation of distillate and bottom streams was observed, with no effect on the quality of the final product [93]. In the other studies, different operating strategies like variable reflux rate are explored to increase the level of terpenic compounds in specific wine distillate fractions to emphasize on floral aroma [63]. A drastic reduction of internal reflux could enhance the recovery of terpenic compounds, while producing a distillate which is rich in floral aromas, and reduction in cooling flow could increase the presence of higher alcohols and esters. The application of MD is compared with VMD, for comparison of volatile composition of wine fractions by two different dealcoholization techniques, i.e., using a membrane contactor (MC) and distillation under vacuum (D). The main difference observed between the two techniques was the concentration grade reached by the dealcoholized fractions which was 5 to 6 times higher when applying VMD, due to associated loss of water [66]. The result obtained was in agreement with previous observations reported in other research works [38]. Recent study, conducted by [89], is concerned with foaming, the main problem associated with stripping which might occur due to formation of gas

bubbles in the liquid and their stabilization through adsorption of surface active agents at their interface. They have studied the application of this technology for fruit juice processing, with the main focus on studying the feasibility of air stripping implementation, using a bubble column for recovery of the flavor components. The summary of the recent research works on application of distillation/stripping technique for flavor recovery in processing liquid foods is assembled in Table 1.

Aroma Recovery Through Centrifugal Distillation

Distillation can be performed in a spinning cone column (SCC), a technology developed by Conetech [24], for recovery of aromas and removing undesirable volatile components from fruit juices and other food liquid streams (see Fig. 3 (part a)). This technology has the advantage that it operates at low temperatures, short residence times, with effective vapor/liquid mixing. Counter-current contacting the vapor and the liquid in alternating and rotating truncated cones which act as contacting stages increases the mass transfer rates and has the advantage over conventional plate columns, operating at atmospheric pressure, since separation efficiency about 20NTU/m, can be achieved in SCC in comparison to 6NTU/m in packed columns [86]. It has been successfully applied for recovery of volatile aroma compounds in wine and beer industry, for removing delicate aromas, removal of sulfur dioxide from grape juice, production of grape concentrates and alcohol reduction in wines [19, 58, 88]. A number of comparative studies are available on the application of this technology for aroma recovery for liquid foods. Table 1 highlights the most recent research conducted on the application of this technology.

Pervaporation Membrane Separation Technique

Pervaporation is an attractive technology for processing thermal sensitive aroma compounds. This membrane process is based on a selective transport of a liquid mixture through a selective ceramic or polymeric membrane [2] (as illustrated in Fig. 3, part a). This technique can be an alternative to conventional separation processes such as steam distillation, liquid solvent extraction, and vacuum distillation and has been successfully applied during the last years, for recovery of aroma compounds from fruits and fruit juices [11, 39, 47, 70, 73, 78] and subsequent addition to the same juice after concentration by evaporation [41, 51, 92]. Pervaporation technique has also been applied for ethanol removal over the last few years [18, 97, 102] and aroma recovery from alcoholic beverages [14, 17, 19, 50]. The most recent studies for the application of this technique, conducted by different researchers in food industry, are summarized in Table 1.

In the recent studies conducted by Catarino et al. [17] and Catarino and Mendes [18] on aroma recovery from beer and

Table 1 Recent research on alternative techniques for recovery of food flavors

| Techniques based on vapor/liquid equilibrium | Flavor type | Mediator | Matrix | Application | Reference |
|-----------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------|--------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|
| Distillation / Stripping | Organic acids, polyphenols, anthocyanins | Polypropylene hollow fibers | Rose wine, Pelaverga, and Barbera red wine | MC & VMD for dealcoholization Influence of treatment on dealcoholized fractions | [66] |
| | Alcohols, esters, acetaldehyde | Membranes, heat processing | Beer and alcoholic drinks | Ethanol removal and flavor recovery through membrane-based and thermal methods | [68] |
| | Terpenes (limonene, linalool, α -terpineol, β -citronellol, geraniol, nerol) | Water vapor/ thermal processing | Wine | Variable reflux rate operating strategies to increase the levels of terpenic compounds in specific distillate fractions to emphasize its floral aroma | [63] |
| | Terpenes (methyl- <i>N</i> -methyl anthranilate, alpha sinensal) | Water vapor | Green mandarin | Vacuum fractional distillation for removal of terpenes | [93] |
| | Ethyl butyrate, hexanal, ethyl acetate, linalool | Air | Fruit juice | Effect of air bubbling on the physicochemical properties of flavors during the extraction of their volatile aroma compounds using a bubble column operated with antifoam | [89] |
| | Maltose, glycerol | Non-porous membrane, TW30–1812-75 (polyamide) | Beer | MD for dealcoholization of beer. Studying effect of vacuum pressure and membrane flux on flavor recovery | [77] |
| | Ethyl acetate, ethanol, butanol, acetaldehyde | Membrane | Fruit juice | Osmotic distillation (OD) and Vacuum membrane distillation (VMD) for aroma recovery. Studying effect of hydrodynamic conditions and vacuum pressure | [45] |
| | Ethyl butanoate, isoamyl acetate, linalool, β -damascenone, furfural, diacetyl, 1,8 cineol, 3-methyl-1butanol, benzaldehyde, cis-hexen-1-ol, 4-terpinenol, eugenol | Sweeping gas, polytetrafluoroethylene (K150) membrane | Berry fruit juice | Sweeping gas membrane distillation (SGMD) for aroma recovery. Studying the influence of temperature, feed and sweeping gas flow rate on recovery | [4] |
| | Ethyl 2,4-decadienoate | Polypropylene (PP) microporous membranes | Fruits (pear) | Pear aroma recovery by VMD | [33] |
| | Ethyl butanoate, isoamyl acetate, linalool, β -damascenone, furfural, diacetyl, 1,8 cineol, 3-methyl-1butanol, benzaldehyde, cis-hexen-1-ol, 4-terpinenol, eugenol | Membrane, water | Black currant juice | Studying VMD for recovery of 12 characteristic aroma compounds | [94] |
| Centrifugal distillation (Spinning cone column SCC) | Ethanol, propanol, isobutanol, amyl alcohol, ethyl acetate, isoamyl acetate, acetaldehyde | Water vapor | Beer | Stripping ethanol and volatile aroma compounds from water vapor stream | [19] |
| | Resveratrol, flavonols (rutin, quercetin, myricetin), | Water vapor | Wine | Dealcoholization of wine, studying antioxidant activity and phenolic | [9] |

Table 1 (continued)

| Flavor type | Mediator | Matrix | Application | Reference |
|-------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------|---------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|
| flavan-3-ols, Anthocyanins, non-flavonoids | | | compound composition of red, rose, and white wine | |
| Ethanol, glycerol, acetate, succinate, acetoin, 2,3-butanediol, acetaldehyde | Membranes, water vapor | Wine | Recovery of aromas in a two-stage process for aroma recovery (28 °C) and for ethanol removal (38 °C) | [88] |
| Alcohols, aldehydes | Membranes, water vapor | Wine | Comparison of SCC technology for aroma recovery and dealcoholization of wine with other technologies | [58] |
| | Polydimethylsiloxane (PDMS) membrane | Wine | PV combined with nano-filtration(NF) for aroma recovery | [84] |
| Hexal, i-AmOL, 1-HexOL, BezAL, BezOL, 2-PhetOL | Polymethylolctylsiloxane–polyetherimide (POMS/PEI) membranes | Beer | Membrane selectivity used in PV from solubility | [29] |
| Isobutanol, ethyl acetate, and isoamyl alcohol | Membranes | Seafood juices | Aroma concentration of fish and shellfish and issues related to technical and economic feasibility of industrial processes | [12] |
| Crustaceans | | | | |
| 2,3-Butanedione, 2,3-pentanedione, 3-methylbutanal, benzaldehyde, acetaldehyde, furfural, 2,5-dimethylpiperazine, 5-methyl furfural | PDMS membrane | Coffee | Membrane in plate and frame module was used to concentrate key aromas of coffee. The effect of feed flow rate, temperature, permeate pressure was investigated | [107] |
| Acetaldehyde, propanol, isobutanol, anyl alcohols (2-methylbutanol plus 3-methylbutanol), ethyl acetate and isoamyl acetate | (POMS/PEI) membrane | Wine | PV combined with nano-filtration (NF) production of dealcoholized wine | [18] |
| Higher alcohols and esters | (POMS/PEI) membrane | Beer | Studying the effect of operating conditions | [17] |
| Isopentyl acetate, 3-methyl-butanol, n-hexanol, and α -ionone | POMS/PDMS membranes | Pomegranate juice | Recovery of aromas | [78] |
| Ethyl acetate, ethyl butyrate, hexanal, limonene, linalool, α -terpineol | PDMS-PVDF-PP composite membrane with a functional layer of PDMS | Orange juice | Recovery of volatile aroma compounds and studying effect of feed flow rate, temperature and permeate pressure | [3] |
| Esters | Dense CO ₂ | Alcoholic beverages | Countercurrent supercritical fractionation (CC-SFF) of flavors in packed columns, membrane contactors and mixer-settler systems | [8] |
| (E)-2-hexenal and hexenal | Supercritical CO ₂ | Apple | CC-SFF of six key apple aromas using dense CO ₂ , studying the effect of temperature, pressure and solvent-to-feed ratio on extraction of aromas | [7] |
| Epicatechin, epigallocatechin, epicatechin gallate, epigallocatechin gallate, caffeine | Supercritical CO ₂ | Green tea | Using different solvents (ethyl acetate, ethyl lactate, and ethanol) with SC-CO ₂ for extraction instatic and dynamic mode in pilot-scale | [105] |
| Fatty acid esters, phenols, cumarin and terpene derivatives | Supercritical CO ₂ | Citrus | Extraction of flavors using ethanol as co-solvent to optimize extraction yield | [100] |

Table 1 (continued)

| Flavor type | Mediator | Matrix | Application | Reference |
|---------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------|--------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------|
| Triolein stearic, oleic, linoleic, linolenic | Supercritical CO ₂ | Vegetable oil | Extraction of vegetable oil from preprocessed seeds, studying the key important parameters for scale-up | [30] |
| Esters (e.g., ethyl acetate, isoamyl acetate, etc.), alcohols (e.g., 2-methyl-1-propanol, n-Butanol, etc.) acids (caprylic acid, isovaleric acid, etc.) | Supercritical CO ₂ | Wine | Aroma recovery from white and red wine using supercritical CO ₂ , studying different ratios of CO ₂ /wine. Recovery of aromas from rose wine in a two-step process | [82] |
| Alcohols (e.g., ethanol, methanol, etc.), esters (e.g., ethyl acetate, isoamyl acetate, etc.) | Supercritical CO ₂ | Grape spirit | Recovery of volatiles using CC-SFE, effect of different solvent-to-feed ratios on extraction yield | [25] |
| Higher alcohols, phenols, fatty acids, esters, ketones | Supercritical CO ₂ | Sugar cane spirits | Extraction of intermediate aromas. Effect of temperature and pressure on extraction yield | [42] |
| Esters, aldehydes, ketones, terpenes, lactones | Supercritical CO ₂ | Brandy | CC-SFE is applied in pilot scale for extraction of aromas and influence of temperature and pressure on extraction yield are investigated | [90] |
| Esters, higher alcohols, diketones, ethanol | Synthetic hydrophobic resins (XAD series and DIAION resins) | Beer | Adsorption of flavor compounds present in beer. Isotherm studies and resin selectivity. Optimum resin proposed for industrial scale. | [83] |
| Phenols | Ion-exchange resins (Amberlyst A26, Amberlite IRA-67) | Olive | recovery of flavors from olive millwaste water. The effect of pH on recovery | [103] [104] |
| Fatty acids (e.g., acetic acid, propionic acid, valeric acid, etc.) | Ion exchange resins (Septra NH2, Amberlyst A21, Septra SAX, Septra ZT-SAX) | Grape | Recovery of volatile flavors, studying effect of pH on adsorption | [80] |
| Carboxylic acids | Amberlite IRA-67, and activated carbon | Fermentation broth | Adsorption from broth under different pH conditions | [109] |
| Phenols | Chitosan (CHI) functionalized Fe3O4 magnetic microspheres coated with polyaniline | Juices | Recovery of flavors | [48] |
| Catechins | Macroporous polymeric resins (XAD resins, DIAION resins) | Tea | Decaffeination of flavors, isotherm studies and resin selection | [65] |
| Benzaldehyde | Activated carbon | Coffee | Fixed-bed adsorption column for recovery of flavors. The effect of feed concentration, flow rate, column diameter and bed length | [32] |

Techniques based on
solid / liquid
equilibrium

wine, the effect of operating conditions such as feed velocity and temperature and permeate pressure are studied on process performance, considering the responses of permeate flux and aromas/ethanol selectivities, ethanol concentration and ratio between higher alcohols and esters in the permeate. They proposed the optimum operating conditions and the range of selectivities for higher alcohols and esters: four alcohols (ethanol, propanol, isobutanol, and isoamyl alcohol), two esters (ethyl acetate and isoamyl acetate) and an aldehyde (acetaldehyde). According to their studies on lab scale, selectivity of higher alcohols was positively affected by the temperature and to a minor extent by the feed velocity, while permeate pressure affects negatively their selectivity due to their low saturated vapor pressures (low volatilities) [17, 56]. This trend was not observed on industrial plant scale. On the other hand, selectivity of esters decreased with temperature and increased with permeate pressure and velocity. As a result, the ratio of higher alcohols/esters increased with the temperature and decreased with feed velocity and permeate pressure. A new industrial process was proposed in further studies for producing non-alcoholic beer [17, 18]. The aroma compounds are obtained by pervaporation of the original beer using the same composite membrane, which they had tested in order to investigate the effect of operating conditions in their previous studies. High permeation temperature and low feed flow rate were the most effective for maximizing the permeation flux and the equilibrium of the flavor profile. For production of dealcoholized wine, they could also successfully combine pervaporation with nano-filtration (NF) for recovery of aroma compounds before the dealcoholization step and adding the recovered aromas back again to the dealcoholized product, which increased the flavor sensation. The application of pervaporation with NF is investigated by Catarino and Mendez [18] and Salgado et al. [84] for recovery of aromas from low-alcohol white wines. They have investigated the performance of the combined units in pilot scale for recovery of aroma components. A two-stage NF process was tested for sugar reduction of must, followed by pervaporation to recover aroma precursors from grape must (i.e., higher alcohols and esters) and restitution of the flavor precursors. They could achieve the best results for obtaining an optimal aroma profile close to original must, by combining pervaporation with a two stage NF. To achieve more desirable results, they proposed the enhancement of mass transfer during pervaporation through increasing the pervaporation time, a higher feed tangential flow or feed pressure which improves the aroma transfer, taking into account the limit for maximum pressure drop. In the other studies conducted by Del Olmo et al. [29], the final quality of the alcohol-free beer was improved through pervaporation to recover the aromas and flavor constituents of beer, such as isobutyl alcohol, ethyl acetate, and isoamyl acetate. The application of pervaporation concentrating volatile aroma compounds in industrial soluble coffee is studied in the research

work conducted by Weschenfelder et al. [107]. They have investigated the effect of feed flow rate, temperature, and permeate pressure on the pervaporation performance of selected compounds in the group of ketones (i.e., 2,3-butanedione and 2,3-pentanedione), aldehydes (i.e., benzaldehyde, and acetaldehyde and furfural and 5-methyl-furfural), and alcohols (i.e., 3-methyl-butanol) and 2,5-dimethylpirazene. For all the tested compounds, permeation flux increased with temperature and results indicated that aroma compound fluxes decreased with partial pressure except for 5-methyl-furfural, and 2,3-butanedione and 2,5-dimethyl pirazene presented the highest enrichment factors in the experimental conditions evaluated in their work. They proposed an optimization step for industrial purposes in order to concentrate the aroma profile for soluble coffee. More information on the current state of research on application of this technology for flavor recovery is given in Table 1.

Aroma Recovery Through Supercritical Fluid Extraction

Supercritical fluid extraction (SFE) is a process which uses substances at pressure and temperature above the critical point (as illustrated in Fig. 3 (part *b*)) as solvents to extract valuable materials [7, 28, 85]. Supercritical extraction with CO₂ has been widely adopted for isolation of volatile aroma compounds in plants and fruits [7, 42, 100] and vegetable oils from preprocessed seeds [30, 69]. There are some research works concerned with aroma recovery from alcoholic beverages [90] combined with a dealcoholization process [15, 64, 82]. Supercritical CO₂ can be applied for batch extraction of solids, for multi-stage counter-current separation and fractionation of liquids, and for adsorptive and chromatographic separations [15, 62]. This technique is mainly carried out at different modes of operation, which is mainly concerned with extraction from solids, carried out in batch or single-stage mode. Single-stage extraction consists of two process steps, extraction and separation of the extract from the solvent. This simple mode of operation enables contacting the feed until a certain mean residual concentration in the solid raffinate is achieved. However, during the extraction process, many factors like extraction kinetics might change due to depletion of the solid substrate from solid that might change the optimum process conditions. In addition, loading the solvents can be enhanced by increasing the number of stages and operating in a counter-current mode. This alteration reduces the amount of solvent required and makes continuous production of extract achievable [15]. Application of counter-current supercritical extraction was studied for apple aroma recovery by Bejarano and Del Valle [7]; the effect of temperature, pressure, and solvent to feed ratio on fractionation and concentration characteristics of six apple aromas is investigated. They could achieve high separation of individual aromas over water, extraction yield of aromas higher than 86%. However, polarity difference

between the tested compounds was the drawback of application of this technique for separation of some tested alcohols from aldehydes. The other recent research work is concerned with extraction of catechins and caffeine from green tea, using different co-solvents (i.e., ethyl lactate, ethyl acetate, and ethanol) and supercritical CO₂ (SC-CO₂) [105]. For the experimental procedure, two different approaches of static (introducing the co-solvent in the extraction cell and pumping SC-CO₂) and dynamic (mixing co-solvent with SC-CO₂ before introduction into the extraction cell) were tested in pilot scale. The highest caffeine extraction yield was obtained with ethyl acetate using both approaches (13 and 14.2 mg g⁻¹ of tea), followed by ethanol (10.8 and 8.8 mg g⁻¹). Lowest extraction yield was achieved using ethyl acetate as co-solvent (lower than 7 mg g⁻¹). Application of ethanol as a co-solvent in extraction of flavors using SC-CO₂ is also investigated for extraction of fatty acid esters, phenols, coumarin, and terpene derivatives from citrus [100]. The most enriched and concentrated extracts of coumarin (osthole) was obtained (approximately 47%) at 170 bar. Furthermore, SCE is successfully applied for flavor recovery and ethanol removal from alcoholic beverages [25, 42, 82, 90]. In the studies conducted by Ruiz-Rodriguez et al. [82], this technique is implemented for aroma recovery and ethanol removal from aqueous solutions. They have developed a two-step process for production of low-alcohol beverage from wine by recovering the aromas in a counter-current packed column using low CO₂/wine ratios. The developed two-step process proved to have similar antioxidant activities and aroma profile to the original wine. Recovery of volatile alcohols and esters is investigated on pilot scale using counter-current supercritical fluid extraction (CC-SFE) from grape spirits [25]. The effect of different solvent-to-feed ratios is examined on recovery of volatiles. As they concluded in their survey, in order to achieve the highest ethanol and volatiles' extraction yield, lowest solvent-to-feed ratio should be used.

SC-CO₂ extraction was employed for extraction of aroma compounds from sugar cane in the work of Gracia et al. [42], for rum production. According to their studies, the extraction yield increased with increasing the temperature and pressure. Optimization of counter-current supercritical fluidic extraction (CC-SFE) conditions is explored by Señoráns et al. [90] for obtaining high-quality brandy aromas. As is demonstrated in their work, increasing the flow rate increased the presence of aroma compounds in the separator. When increasing the extraction pressure, a higher sample flow rate has to be used to achieve the maximum extraction.

Supercritical CO₂ technology is adopted widely and its economic feasibility and advantages over conventional techniques should be proven for each applied technology. Despite initial high capital costs, operating costs would be lower, as it is operated as a continuous process [62, 74, 82], and overall feasibility can be proven at certain scales of operation. This

technology enables the possibility of combining an extraction operation with column fractionation under supercritical conditions to concentrate the bioactive flavor components [62]. In comparison to other techniques for aroma recovery, less attention has been paid to application of this technology for recovery of aroma compounds from liquid food streams. Further studies on application of this technique for aroma recovery is recommended, especially for production of alcoholic beverages, which is of high economic interest [10].

Regeneration and Recovery of Aromas via Adsorption

Among the available techniques for aroma recovery, adsorption is a technique which shows potential for selective recovery of the flavor compounds and can be applied as an alternative to thermal processes or can be combined with distillation/stripping in an integrated process [22, 46, 72]. It can be applied as a technique for selective recovery of the compounds based on their affinity toward a ligand (affinity chromatography), based on charge (ion-exchange chromatography), hydrophobicity (hydrophobic interaction chromatography (HIC)), and based on polarity, or size of the molecules (size exclusion chromatography (SEC)) [43, 44, 87]. The mechanism of different modes of separation in adsorption technique is depicted in Fig. 3 (part c). During the last 2 years, this technique has been successfully applied to recover mainly phenolic compounds besides other volatile aroma components from liquid streams in food processing industry using adsorbents such as activated carbon, chitosan, minerals (zeolites), and synthetic resins [16, 32, 48, 65, 83, 103, 104, 108]. In the recent studies, application of this technique is investigated for recovery of coffee aroma compound benzaldehyde on granular activated carbon derived from coconut husk [32]. The effect of fixed-bed operating parameters like inlet concentration and inner diameter of the bed are investigated on adsorption and recovery of the aroma component. They could use the obtained results from column performance to perform a scale-up study with error of less than 12%. The current research focus is on development of this technique to synthesize new adsorbent materials which have more affinity to adsorb aroma components [48]. The application of synthesized chitosan, functionalized with Fe₃O₄ magnetic microspheres coated with polyaniline, is studied for adsorption of phenolic components in juice samples. According to the obtained results, synthetic microspheres showed high permeability and acceptable recovery of the phenolic components (between 85 and 107%) [48]. Considering the high potential of this technique for aroma recovery, in combination with other separation techniques or as an alternative, further research is worthwhile to investigate new synthesized and functionalized adsorbent materials which are also applicable in food industry for recovery of volatile aroma components.

Concluding Remarks

Various techniques are proposed and tested according to studies reported in literature for recovery of aroma components, which all aim for minimizing the loss of aroma compounds and recovering the key components which are valuable in producing a high-quality final product. The technologies that can be applied for aroma recovery in food industry according to former investigations are stripping or distillation, which can be performed as membrane vacuum distillation or centrifugal distillation, pervaporation, supercritical extraction, and adsorption. Among these available techniques, stripping and distillation are widely applied for aroma recovery in processing alcoholic beverages and juices. Pervaporation as an alternative technique could show promising achievements for recovery of the aroma compounds from aqueous food streams. The current research focus on the application of this technique on aroma recovery is on the optimization of conditions to enhance the selectivity over specific aroma components in the process. In comparison to the other alternatives, less attention has been paid to supercritical extraction of aromas, specifically for aroma recovery from alcoholic beverages. The great selectivity of supercritical extraction has been proved by several investigators, which are demonstrative, since they fully take profit of applying supercritical fluid carbon dioxide as a non-toxic, and natural GRAS (Generally Recognized as safe) solvent with high selectivity at relatively low temperature, which prevents alteration of thermolabile products. Applying this technology, selectivity, and capacity can be tuned by changing operating pressure and temperature. Meanwhile, combining this technique with counter-current flow and reflux in a multistage contactor can lead to an optimized process conditions. Further research is recommended to study the application of this technique for recovery of aroma compounds, especially in alcoholic beverage industry which is of high economical interest, and where alcoholic beverage fractionation is a challenge, since ethanol is present at significant concentration in comparison to aroma components which are often present at trace levels and modifies the carbon dioxide solvent power in reducing its selectivity over water and other aroma products. Among the reviewed techniques, adsorption can be applied as a promising technique for selective recovery of aroma components and adding back the recovered key components to process streams, in order to produce a high-quality final product. Additional research is required to study the possibilities of applying this technique for flavor recovery as an alternative or combined with thermal processing.

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