

## CHAPTER 2

# PROCESSING OF FRUITS: AMBIENT AND LOW TEMPERATURE PROCESSING

### 2.1. FRUIT PRODUCTS AND MANUFACTURING PROCESSES

World trade of fruit and vegetable juice averaged nearly US\$4,000 million last decade (FAOSTAT, 2005). By far the largest volume of processed apples and oranges, the two most important fruit commodities, is in the form of juices, and a great part of the present chapter is devoted to describing the processing of these liquid foods. There are however many other products obtained from fruits, including canned, dried, and frozen fruit; pulps; purées; and marmalades. Table 2.1 lists final products and processes applied on selected fruits. In addition, developments in aseptic processing have brought new dimensions and markets to the juice industry.

Juices are a product for direct consumption and are obtained by the extraction of cellular juice from fruits; this operation can be done by pressing or diffusion. Fruit juices are categorized as those without pulp (“clarified” or “not clarified”) and those with pulp (“pulp,” “purées,” and “nectars”). Other classifications include “natural juice” products obtained from one fruit, and “mixed juice” products obtained from the mixing of two or three juices of different fruit species or by adding sugar. Juices obtained by removal of a major part of their water content by vacuum evaporation or fractional freezing are defined as “concentrated juices.”

Fruit composition is mainly water (75–90%), which is mainly found in vacuoles, giving turgor (textural rigidity) to the fruit tissue. Juice is the liquid extracted from the cells of mature fruits. Fruit cell wall is made of cellulose, hemicellulose, pectic substances, and proteins.

The primary cell wall, composed of crystalline cellulose microfibrils, is made up of polymers of b-D-glucose linked by b-1-4-glycosidic linkages and cellulose embedded in an amorphous matrix of pectin and hemicelluloses. The definition of a mature fruit varies with each type. Typically, sugar and organic acid levels, and their ratio indicate maturity stage. The extracted liquid is composed of water, soluble solids (sugars and organic acids), aroma and flavor compounds, vitamins and minerals, pectic substances, pigments, and, to a very small degree, proteins and fats. The various sugars, such as fructose, glucose, and sucrose, combined with a large number of organic acids (most important being citric, malic, and tartaric), help give the fruit its characteristic sweetness and tartness.

During ripening of fruits, a general decrease in acidity and starch as well as an increase in sugars is seen. Moreover, formation of odors, breakdown of chlorophyll, and hydrolysis of pectic substances also occur. It must be noted that plant tissues continue to ripen after harvest. Finally, senescence occurs, at a rate accelerated by the increase in ethylene.

**Table 2.1.** Principal fruit products and manufacturing processes.

Product	Fruit	Process description
Canned	Apples	Canned apple is the product prepared from fresh apples of one variety, which are not overripe, and whose fruit is packed with or without any of the following ingredients: water, salt, spices, nutritive sweetening ingredients, and any other ingredients permissible under regulations. The product is then heat processed to ensure preservation in hermetically sealed containers
	Apple sauce	Canned apple sauce is the product prepared from comminuted or chopped apples, which may or may not be peeled and cored, and to which may have been added thereto one or more of the optional ingredients specified by regulations. The product is heated and, in accordance with good manufacturing practices, bruised apple particles, peel, seed, core material, and other coarse, hard, or extraneous materials are removed. The product is processed by heat, either before or after sealing, so as to ensure preservation. The soluble solids' content is $\geq 9^\circ\text{Brix}$
	Cranberry sauce	Canned cranberry sauce is the jellied or semijellied cranberry product prepared from clean, sound, matured cranberries, and contains sweetening ingredients and water. Pectin may be added to compensate for deficiency of the natural pectin content of the cranberries. The mixture is concentrated and sufficiently processed by heat to ensure preservation of the product. Final soluble solid is $\cong 35\text{--}45\%$
	Fruit salads	Canned fruits for salad consist of carefully selected apricots, cherries, yellow clingstone peaches, pears, pineapple, and grapes. The product is packed in a suitable liquid medium with or without the addition of sweetening ingredients, or other permissible ingredients. The product is heat processed and is processed to ensure preservation of the product in hermetically sealed containers
Frozen	Apples	Frozen apples are prepared from fresh apples of one variety, not overripe, which are peeled, cored, trimmed, sliced, sorted, washed, and properly drained before filling into containers. Sweetening ingredient and any other ingredient permissible under regulations may be used. The product is frozen in accordance with good commercial practice and maintained at temperatures necessary for the preservation of the product
	Apricots	Frozen apricots are prepared from fresh fruit of one variety, which are not overripe, which are sorted, washed, and may be trimmed to ensure a clean and wholesome product. The apricots are properly drained of excess water before placing into containers. The addition of sweetening ingredients, including syrup containing pureed apricots, suitable antioxidant ingredients, and or any other ingredients permissible under regulations is allowed
	Berries	Frozen berries are prepared from properly ripened fresh fruit berries, are stemmed and cleaned, may be packed with or without packing media, and are frozen and stored at temperatures necessary for the preservation of the product. The same is applicable to frozen blueberries. Frozen cranberries do not need stemming before freezing
Frozen	Cherries	Frozen sweet cherries are prepared from fresh fruit of one variety, which are not overripe, fruit of any commercial variety of sweet cherries, which are sorted, washed, and drained. The addition of nutritive sweetening ingredients is allowed. The product is frozen in accordance with good commercial practice and maintained at temperatures necessary for the preservation of the product
	Grapefruit	Frozen grapefruit is prepared from fresh fruit of one variety, which are not overripe. After the fruit has been washed and peeled, and separated into segments by removing the core, seeds, and membrane it is packed with or without packing additives. The product is frozen and stored at temperatures necessary for the preservation of the product
	Lemon	Frozen lemon concentrate is the product prepared from lemon juice (from fresh, sound, ripe, and thoroughly cleansed fruit) and lemonade ingredients (sweeteners; lemon oil, its extracts, or emulsions) and water in sufficient quantities to standardize the product. The product contains $\geq 48.0^\circ\text{Brix}$ (corrected for acidity). Such juices may be fresh or frozen, or fresh concentrated or frozen concentrated; processed in accordance with good commercial practice and is frozen and maintained at temperatures sufficient for the preservation of the product

*(continued)*

Table 2.1. (Continued)

Product	Fruit	Process description
Dried	Melon	Melon balls are spheres of melon flesh prepared from balls of suitable varieties of sound, fresh melons. The balls are prepared and washed in a manner to assure a clean and wholesome product. The product may be packed with the addition of a suitable fruit and or vegetable garnish; nutritive or non-nutritive sweetening ingredients, including syrup and any other ingredient permissible under regulations. It must be frozen in accordance with good commercial practice and maintained at temperatures necessary for preservation
	Peaches	Frozen peaches are prepared from fresh peaches of one variety, which are not overripe, peaches are peeled, pitted, washed, cut, and trimmed to assure a clean and wholesome product. The peaches may be packed with the addition of a sweetening ingredient, including syrup and/or syrup containing pureed peaches and any other permissible ingredients. The product must be frozen in accordance with good commercial practice and maintained at temperatures necessary for the preservation
	Pineapple	Frozen pineapple is prepared from the properly ripened pineapple fruit, which is peeled, cored, trimmed, and washed; is packed with or without packing media; and is frozen and stored at temperatures necessary for the preservation of the product
	Plums	Frozen plums are prepared from clean, sound, fresh fruit of any commercial variety of plums, which are sorted, washed, drained, and pitted; which may be packed with or without the addition of a nutritive sweetening ingredient; and which are frozen in accordance with good commercial practice and maintained at temperatures necessary for the preservation of the product
	Apples	Dried apples are prepared from fresh apples of one variety, which are not overripe, by washing, sorting, trimming, peeling, coring, and cutting into segments. The prepared apple segments are properly dried to remove the greater portion of moisture to produce a semidry texture. The product may be sulfured sufficiently to retard discoloration. The sulfur dioxide content of the finished product should not exceed 1,000 parts per million. No other additives are allowed
	Apricots	Dehydrated low-moisture apricots are prepared from fresh fruits of one variety, which are not overripe, which are cut, chopped, or otherwise prepared into various sizes and shapes; are prepared to assure a clean, sound, wholesome product; are processed by dehydration whereby practically all of the moisture is removed to produce a very dry texture; and are placed in a container, which has low moisture. The product is packaged to assure dryness retention and should be sulfured at a level sufficient to retain a characteristic color
	Figs	Dried figs are prepared from clean and sound fruits and are sorted and thoroughly cleaned to assure a clean, sound, wholesome product. The figs may or may not be sulfured, or otherwise bleached
	Peaches	Dried peaches are the halved and pitted fruit from which most of moisture has been removed. The dried fruit is processed to cleanse and it may be sulfured sufficiently to retain color
	Pears	Dried pears are made with the halved fruit, which may or may not be cored, from which the external stems and calyx cups have been removed. Before packing, the dried fruit may be sulfured sufficiently to color
	Raisins	Processed raisins are dried grapes of vinifera varieties, such raisins as Thompson Seedless Sultanian, Muscat of Alexandria, Muscatel Gordo Blanco, Sultana, or White Corinth. The processed raisins are from fresh fruit, which are not overripe. Grapes are properly stemmed and cap stemmed, seeded, sorted or cleaned, or both, and are washed in water to assure a wholesome product
	Prunes	Dehydrated prunes are prepared from clean and sound prunes, which are pitted and prepared into various sizes and shapes, washed, and processed by dehydration to produce a very dry texture. The product is then packaged to assure retention of the dryness characteristic of the product. A safe preservative may be added

(continued)

Table 2.1. (Continued)

Product	Fruit	Process description
Juices	Grape	Frozen concentrated sweetened grape juice is prepared from concentrated unfermented single-strength grape juice from fresh fruit, which are not overripe, with or without aging, or grape juice depectinization, and is then concentrated. Single-strength grape juice or natural grape essence, or a combination of single-strength grape juice and natural grape essence may be mixed to the concentrate and may or may not be packed with the addition of ingredients like sweeteners, edible fruit acid, and ascorbic acid. The product is then frozen in accordance with good commercial practice
Juices	Apple frozen concentrate	Frozen concentrated apple juice is prepared from the concentrated unfermented, liquid obtained from apple juice during the first pressing of properly prepared, clean, mature, fresh apples by good commercial processes. The juice is clarified and concentrated to at least 22.9° Brix. The apple juice concentrate so prepared, with or without the addition of ingredients permissible under regulations, is packed and frozen in accordance with good commercial practice and maintained at temperatures necessary for the preservation
	Canned apple juice	Canned apple juice is the unfermented juice obtained from sound, ripe apples, with or without parts. No water may be added directly to the finished product. However, concentrated apple juice is allowed. Apple essence may be restored to a level that provides a natural apple juice flavor
	Canned grape juice	Canned grape juice is the unfermented liquid obtained from the juice of properly matured fresh grapes. Such grape juice is prepared without concentration, without dilution, is packed with or without the addition of sweetening ingredients, and is sufficiently processed by heat to assure preservation of the product in hermetically sealed containers
	Lemon single-strength	Canned lemon juice is the undiluted, unconcentrated, unfermented juice obtained from sound, mature lemons of one or more of the high-acid varieties. The fruit is prepared by washing prior to extraction of the juice to assure a clean product. The product is sufficiently processed with heat to assure preservation in hermetically sealed containers
	Lemon concentrate	The fruit is prepared by sorting and by washing prior to extraction of the juice. The concentrated lemon juice is prepared and concentrated in accordance with good commercial practice. It may or may not require processing by heat, subsequent refrigeration, or freezing to assure preservation of the product. The finished product may contain added pulp, lemon oil to standardize flavor, and or permissible chemical preservatives.
	Tangerine	Concentrated tangerine juice is the tangerine concentrated product obtained from sound, mature fruit. The fruit is prepared by sorting and by washing prior to extraction of the juice. The concentrated tangerine juice is processed in accordance with good commercial practice, and may or may not require processing by heat or subsequent refrigeration to assure preservation of the product. Cold-pressed oil to standardize flavor and permissible chemical preservatives may be added
Others	Marmalade	Orange marmalade is the semisolid or gel-like product prepared from orange fruit ingredients together with ingredients like sweeteners, food acids, food pectin, lemon juice, or lemon peel. Soluble solids of finished marmalade is $\geq 65\%$

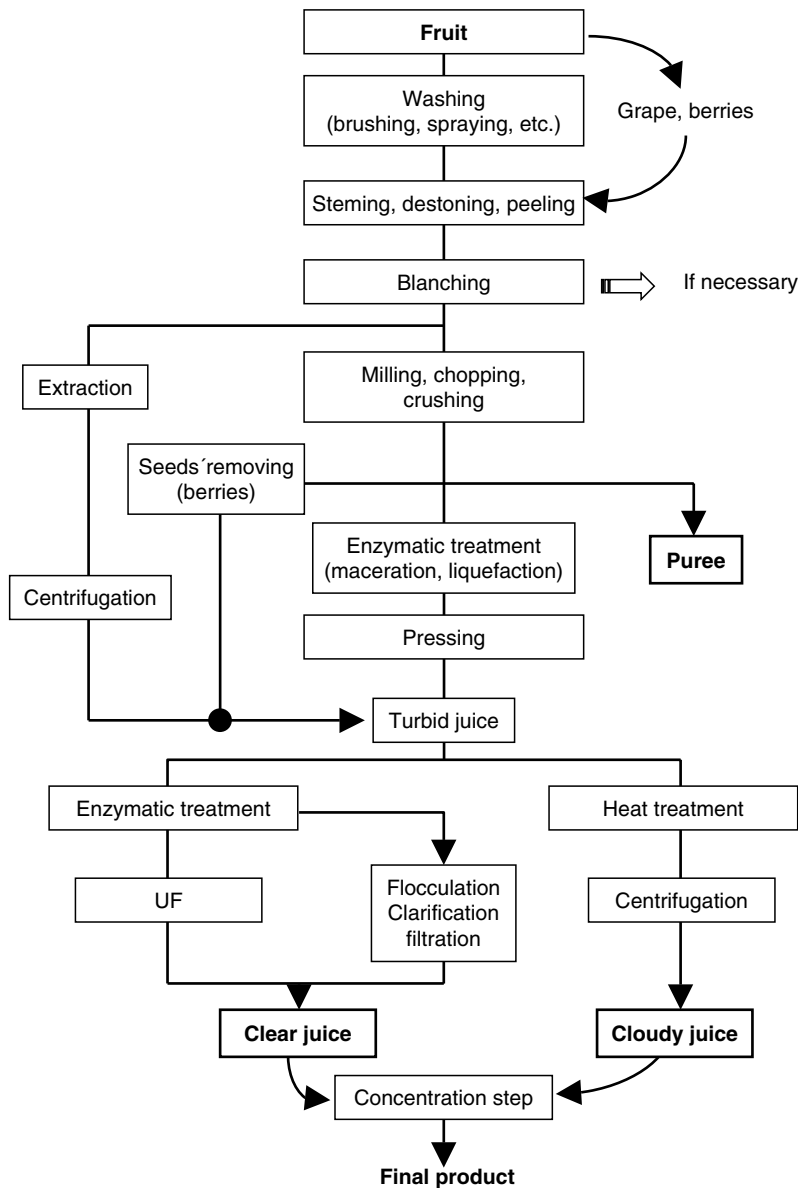
Source: Hui (1991); Nagy et al. (1992); Somogyi et al. (1996).

## 2.2. FRUIT JUICE AND PULP PROCESSING

Fruit processing plants can vary from a simple facility for single juice extraction and canning, to a complex manufacturing facility, which has ultrafiltration and reverse osmosis equipment, cold storage, and waste treatment plant. A simplified characteristic flow diagram of a juice

processing line is shown in Fig. 2.1. Processed products can be either single strength or bulk concentrate, and are available either as clarified or cloudy juice. Production of fruit juices can be divided into four basic principal stages:

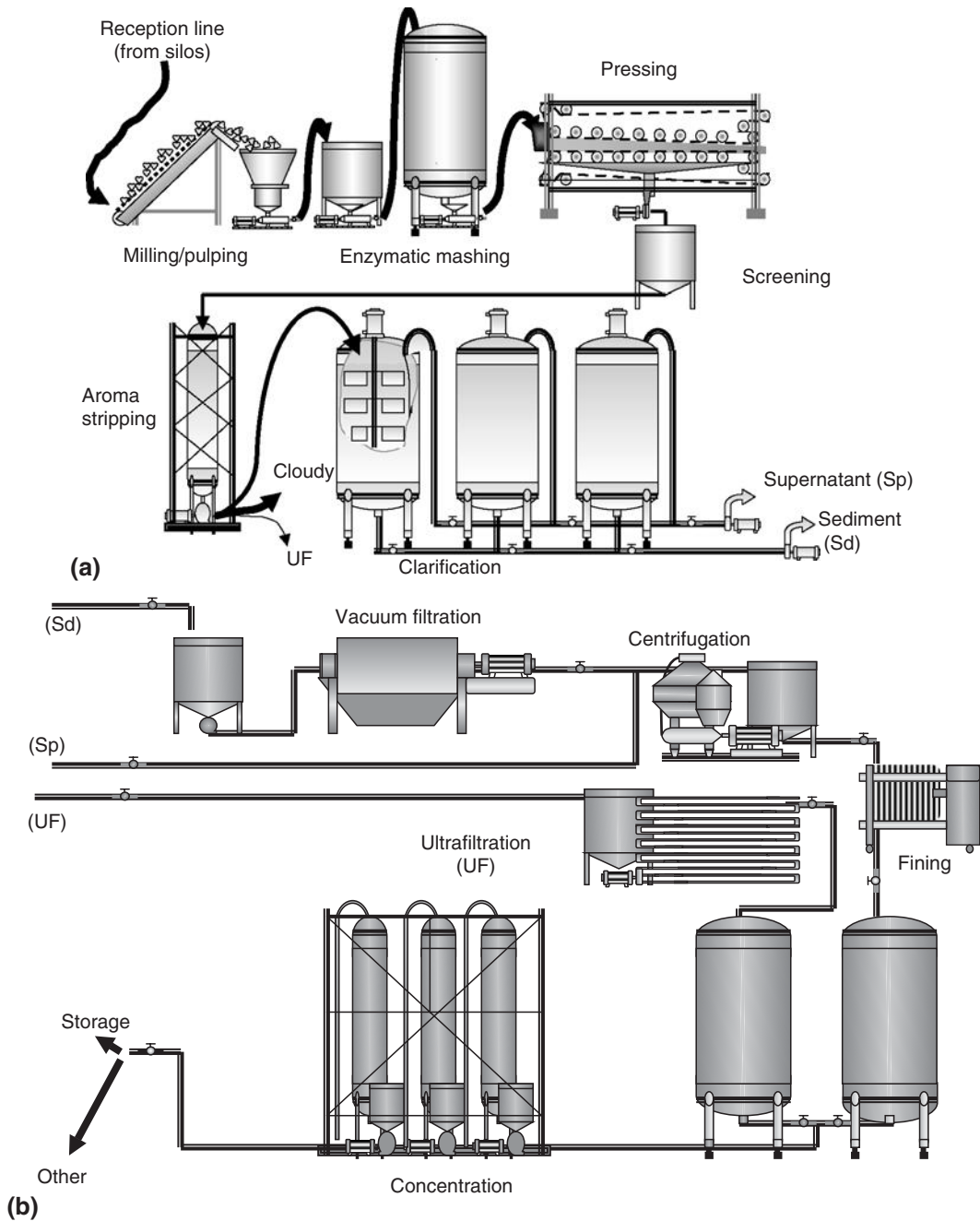
- Front-end operation
- Juice extraction



**Figure 2.1.** Typical fruit juice (clear or cloudy) and purée-processing line steps.

- Juice clarification and refining
- Juice pasteurization and concentration.

Figure 2.2a and b shows descriptive sketches of alternative processing steps for cloudy and/or clarified apple juice concentrate elaboration.



**Figure 2.2.** Typical apple juice processing plant. (a) From fruit to cloudy juice; (b) From cloudy juice to concentrate.

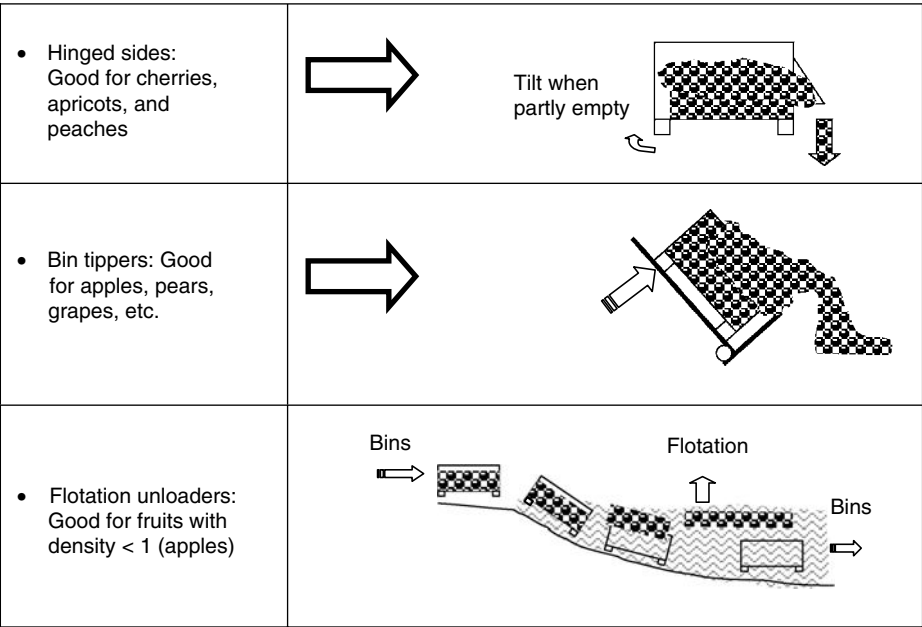
2.2.1. Front-End Operations

This stage includes those operations related with the reception and classification of fruits in the manufacturing plant:

2.2.1.1. Reception Line

- *Weighing of incoming fruit:* Origin and variety are usually recorded in this step.
- *Unloading of fruits into silos system:* Harvesting containers known as bins are commonly used worldwide for transportation of fruits from the orchard to the processing plant. Standard bins are  $1.21 \times 1.21 \times 1.0$  m in size. Up to 30 or more bins may be placed in a single truck. Once in the plant, bin dumping–unloading can be performed at least in three different ways, depending on the fruit (Fig. 2.3).
- *Sampling and laboratory testing:* Table 2.2 lists the recommended fruit controls at the reception in processing plant, including assay of soluble solids, yield, Brix–acid ratio, etc. Other special tests are Magnus–Taylor pressure tester for pears and apples, and background color for peaches.
- *Washing of fruit:* The harvested fruit is washed to remove soil, microorganisms, and pesticide residues. Spoiled fruits should be discarded before washing in order to avoid contaminating the washing tools and/or equipment and the contamination of other fruits during washing. Washing efficiency can be estimated by the total number of microorganisms present on fruit surface before and after washing.

Apples require heavy spray applications and rotary brush wash to remove any rot. Many fruits such as mechanically harvested berries are air cleaned on mesh conveyors or vibrators



**Figure 2.3.** Unloading of fruits into silos systems. Reprinted from the Encyclopedia of Food Science and Nutrition. Lozano J.E., Separation and Clarification, pp. 5187–5196 (copyright) 2003, with permission from Elsevier.

**Table 2.2.** Recommended fruit controls at reception.

Checks per lot	Checks for every 10 lots	Once during harvest season
Color	Density	Ascorbic acid
Taste	Water content	Mineral substances
Texture	Total sugars, reducing sugars	Tannic substances
Flavor	Total acidity	Pectic substances
Soluble solids (°Brix)		
Variety		
Sanitary evaluation		

passing over an air jet. Washers are conveyor belts or roller conveyors with water sprays, reel (cylinder) type with internal spray (Fig. 2.4), brushes and/or rubber rolls with or without studs. Vibratory-type washers are very effective for berries and small fruits. Brushes are effective in eliminating rotten portions of fruits, thus preventing problems with micotoxins (patulin in apples). Some usual practices in fruit washing are:

- Addition of detergents or 1.5%-HCl solution in washing water to remove traces of insecticides and fungicides;
- Use of warm water (about 50°C) in the prewashing phase;
- Higher water pressure in spray/shower washers.

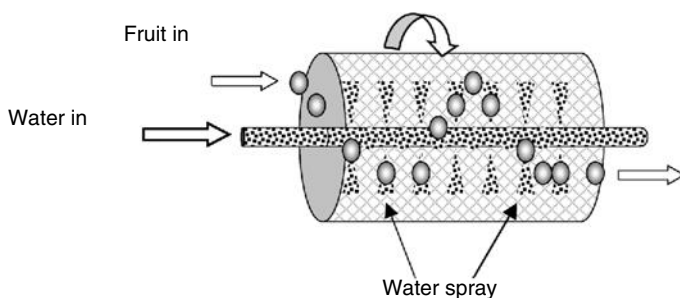
Washing must be done before the fruit is cut in order to avoid losing high-nutritive value soluble substances (vitamins, minerals, sugars, etc.).

#### 2.2.1.2. Final Grading, and Inspection and Sorting

Fruit sorting covers two main separate processing operations:

- (1) Removal of damaged fruit and any foreign substance; and
- (2) Qualitative sorting based on organoleptic criteria and maturity stage.

The most important initial sorting is performed for variety and maturity. However, for some fruits and in special processing technologies, it is advisable to carry out a manual dimensional



**Figure 2.4.** Sketch of a reel washer with internal spray. Reprinted from the Encyclopedia of Food Science and Nutrition, Lozano J.E., Separation and Clarification, pp. 5187–5196 (copyright) 2003, with permission from Elsevier.



**Table 2.3.** Fruit sorting methods.

Sorting method	Description
By size	Rollers (cherries), diverging belts, reels with holes
By weight	Apples and citrus sorters. Sort into 20 or more weight grades
By texture firmness	Bounce system (cranberries)
By color	Citrus color sorter measures green to yellow ratio

sorting (grading). Sorting may be performed by different ways, such as those listed in Table 2.3 (Fellows, 1988):

- *Aligning*: Feeding into some processes (peeling, trimming, etc.) needs the fruit to be placed in a single line. This may be performed with accelerating belts or water flumes.
- *Peeling (skin removal)*: Although manual peeling is still used for certain large vegetables, the method is very expensive. When required, fruits are usually peeled with one of the methods (Woodroof, 1986; Fellows, 1988) listed in Table 2.4. In general, loss increases with surface to volume ratio and decreases with fruit size. Mechanical methods are the worst, with up to 30% loss, while chemical (caustic) methods reduce loss to  $\leq 10\%$ .
- *Trimming*: This is usually a manual operation that precedes cutting, in order to eliminate few defective pieces.
- *Cutting*: Many special cutters are available, including sector cutters for apples, berry slicers, dicers, etc.
- *Pitting and coring*: This operation usually occurs after sorting and peeling. In peaches, pitters cut away some flesh. Automatic cherry pitters have also been developed.
- *Belt conveyor*: Transport fruits to juice extractors (citrus), crusher and mills (pomes), or stem and seed remover (grapes and berries).

**Table 2.4.** Peeling methods.

Method	Description
Mechanical peeling	<ul style="list-style-type: none"> <li>• <i>By abrasion</i>: It is used in batch with rotating abrasive base and water wash. This method is inefficient, with excessive losses.</li> <li>• <i>Abrasive roll peelers</i>: This is a continuous method that combines rolls and brushes.</li> <li>• <i>Blade type</i>: The fruit rotates and mechanized knives separate the peel.</li> <li>• <i>Live knife</i>: Incorporates hydraulic control of the knife pressure. Good for apples and pears.</li> </ul>
Steam peeling	<ul style="list-style-type: none"> <li>• Pressure steam peeling make the peel blow off with pressure drop coming out of peeling chamber. May be combined with dry caustic peeling system.</li> </ul>
Chemical peeling	<ul style="list-style-type: none"> <li>• Caustic peeling is extremely common. The simplest type involves immersion on a pocketed paddle wheel, with hot NaOH (20%), followed by scrubbing and washing. Tomatoes, peaches, and apples are peeled by this method. KOH is preferred because of its tissue penetration and disposal properties.</li> </ul>
Hot gas peeling	<ul style="list-style-type: none"> <li>• When hot gas contacts a vegetable on the belt or roller conveyor, the skin is blown off by the steam formed. It is generally not used in fruits.</li> </ul>
Freeze-thaw peeling	<ul style="list-style-type: none"> <li>• Fruit is frozen in a low temperature medium (<math>-40^{\circ}\text{C}</math>) for few seconds and then warmed in water (<math>40^{\circ}\text{C}</math>). As a result of freezing the immediate subpeel cells are disrupted, releasing pectinases, which free the peel. Peeling loss is reduced to a minimum.</li> </ul>

### 2.2.2. Extraction

The method of separating most of water and soluble solids (juicing) depends on the fruit variety.

#### 2.2.2.1. Citrus

There are three main types of extractors manufactured by different companies (Ramaswamy and Abbatemarco, 1996):

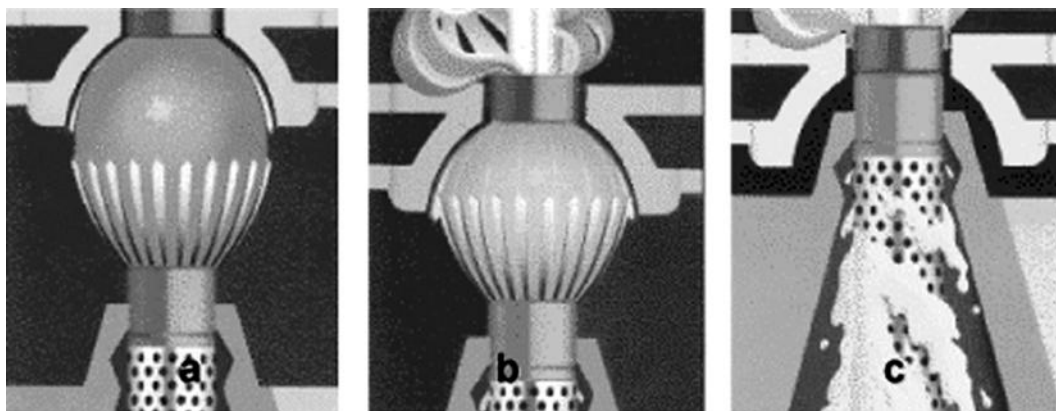
- (1) The *FMC citrus juice extractor*, in which juice is extracted from the whole fruit without first cutting the fruit into half. Outlet streams carry juice peel, center part, and oil emulsion.
- (2) The *Brown extractor*, in which the fruit is cut into half. Outlet streams are juice of high yield and quality, and rag and peel.
- (3) The *Rotary press*, in which the fruit is cut in half and the juice extracted in rotary cylinders.

More than 75% of the world's processors use FMC technology, this process is described in more detail here.

When the upper and lower cups start to come closer to each other, the upper and lower cutters cut two holes in the fruit (Fig. 2.5a). As the upper and lower cups continue to come together, the peel is separated from the fruit (Fig. 2.5b). The peeled fruit moves into the strainer tube where the juice is instantaneously separated from the seeds and the rest of the fruit (Fig. 2.5c).

#### 2.2.2.2. Pomes

There are few problems in reducing the size of fresh “hard” apples or pears. After washing, pome fruits are milled. The fruit to be milled is continuously fed into the milling device. For the disintegration fixed positioned or rotating grinding knives may be used. Depending on the product quality different types of knives need to be selected. The types of fruit mills generally used are:



**Figure 2.5.** FMC citrus juice extractor (with permission).

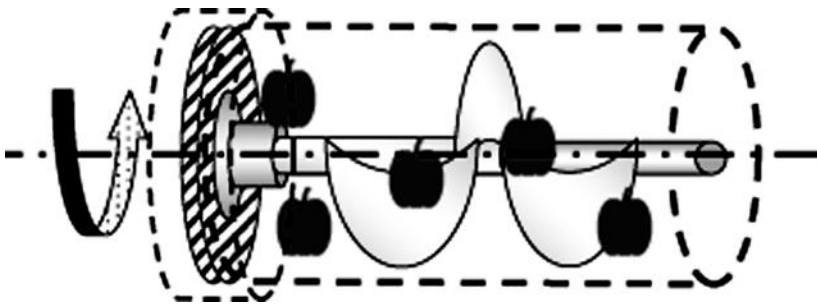


Figure 2.6. Fruit grinding mill.

- *Fruit grinding mill*: The milling tool is a rotating disk with radially arranged grinding knives. The speed of the disk is variable, permitting to produce the required particle size (Fig. 2.6).
- *Rasp or grater mill*: It consists of a revolving metal cylinder with adjustable toothed blades, which rotate toward a set of parallel metal knives or plates.
- *Fixed blade hammer mills*: The rotor with fixed blades rotates within a perforated screen. Hammer mills may be horizontal, sloping, or vertical shaft mounting (Fig. 2.7).

Mills must not produce too much *fines* as these will contribute to pressing and later high pulp content in the juice. The particles should all be about the same size. Grater mills are found to be more efficient with firm fruits, while hammer mills are more suited for mature or softer fruits, provided speed is properly adjusted.

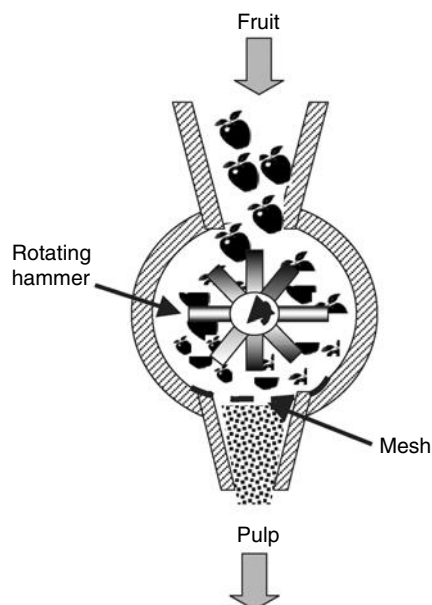


Figure 2.7. Hammer mill.

### 2.2.2.3. Pressing

Most systems for extracting juices from apples and similar fruit pulps use some method of pressing juice through cloth of various thicknesses, in which pomace is retained. These systems, called filter presses, include (Lozano, 2003): (i) rack and cloth press, (ii) horizontal pack press, (iii) continuous belt press, and (iv) screw press.

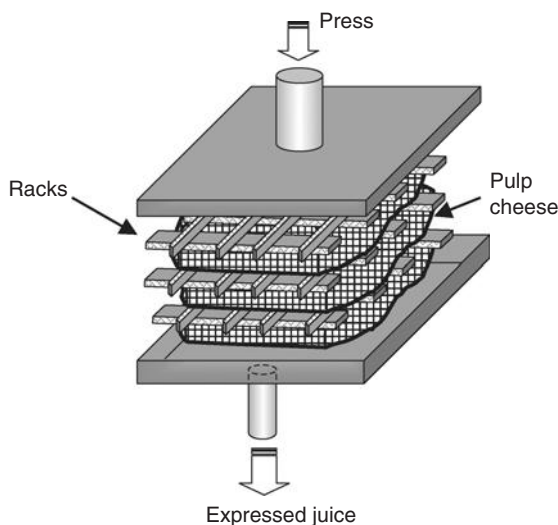
- (1) In a *rack and cloth press* the milled fruit pulp is placed in a nylon, Dacron, or polypropylene cloth to form a “cheese,” with the help of a cheese form. Layers of up to 10-cm thick pulp cheeses, separated by racks made of hardwood or plastic, are stacked up to 1 m or more in height depending on maturity of the fruit and size of racks (Fig. 2.8).

Rack and cloth presses are efficient but very labor intensive as they require operation, cleaning, and repairing. Maximum yield may be obtained by use of a series of two to three pressure heads located around a central pivot, using pressures up to 200 atm.

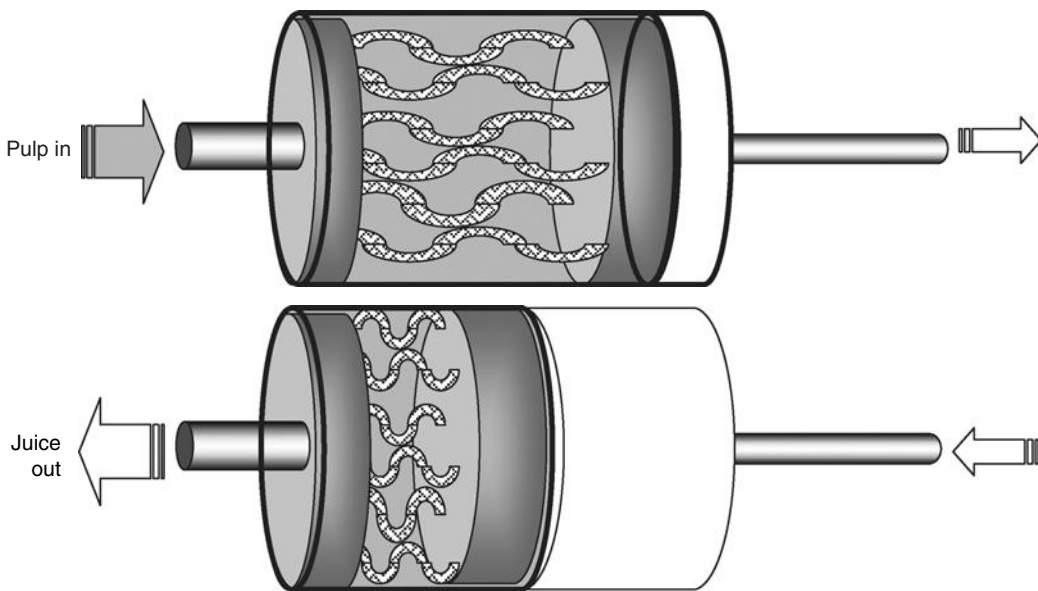
- (2) Cage presses are horizontal presses with enclosed cages of several cubic meters in which pressing takes place. Pomace is pumped into the cage without contact with air, thereby reducing oxidation (Fig. 2.9a). The cage is filled with a complex filter systems consisting of grooved flexible rods filled with sleeves of press cloth material.

During the pressing step (Fig. 2.9b), the juice passes from the pulp, through press cloth sleeves, along grooves in the flexible rods, and out to collecting channels at the ends of the cage and the piston.

The drum may be rotated, thereby breaking up the pulp and adding more water. This permits a second pressing with more juice extraction. The whole process may be automated. Although some cleaning labor is saved, rods and sleeves require a considerable amount of



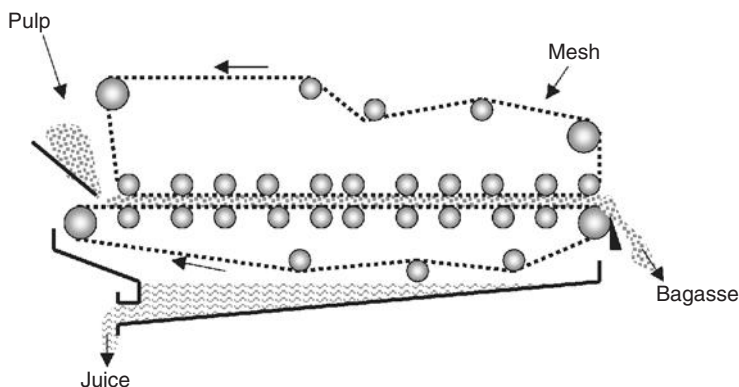
**Figure 2.8.** Sketch of a rack and cloth press. Reprinted from the Encyclopedia of Food Science and Nutrition, Lozano J.E., Separation and Clarification, pp. 5187–5196 (copyright) 2003, with permission from Elsevier.



**Figure 2.9.** Hydraulic press: (a) loading, (b) pressing.

maintenance. These presses may slow down the operating cycle for production of stable cloudy nonoxidized juice.

- (3) *Continuous belt press*: Based on the Ensink design for paper pulp pressing this type of presses offers a truly continuous operation (Fig. 2.10). In belt presses, a layer of mash (pulp) is pumped onto the belt entering the machine. The press aid may be added for improved yield.
- (4) *Screw presses*: A typical screw press consists of a stainless steel cylindrical screen, enclosing a large bore screw with narrow clearance between screw and cylinder. Adjustable back-pressure is usually provided at the end of the chamber. Breaker bars must be incorporated to disrupt the compressing mash. Capacity for screw presses of 41-cm diameter is up to 15,000 kg/h (Bump, 1989).



**Figure 2.10.** Sketch of a typical fruit belt press.

#### 2.2.2.4. Other Extraction Methods

- *Centrifugation*: Both cone and basket centrifuges have been used in producing fruit juice. Both systems have resulted in high levels of suspended solids and a high investment cost for a given yield. Horizontal decanters are presently used for juice clarification.
- *Diffusion extraction*: This was adapted from the method used for the extraction of sugar from sugar beet. Extraction is a typical countercurrent-type process. It is desirable to retain the same driving force  $\Delta C$  (concentration of soluble components in solids versus concentration of soluble components in liquids). In order to maintain a constant  $\Delta C$  throughout the extraction process, it is necessary to carry out a continuous weighing of ingoing apple slice and control the water flow to the counterflow extractor, by means of a relatively simple control loop (Fig. 2.11).

The diffusion extraction process is influenced by a number of variables, including temperature, thickness, water, and fruit variety. Slices from extractors pass through a conventional press system, and the very dilute juice is returned to the extractors. It is seen that the extra juice yield from diffusion extraction compensates the extra energy cost involved for concentration.

- *Addition of press aids*: Hydraulic pressing does not usually require addition of press aids, unless exceptionally overmature fruit is used. For continuous screw presses however it is usually necessary to add 1% (w/w) or more of cellulose. Mixing of cellulose and fruit occurs in the mill and subsequent pumping to press. Pumping is commonly performed with a Moyno-type moving cavity food pump.

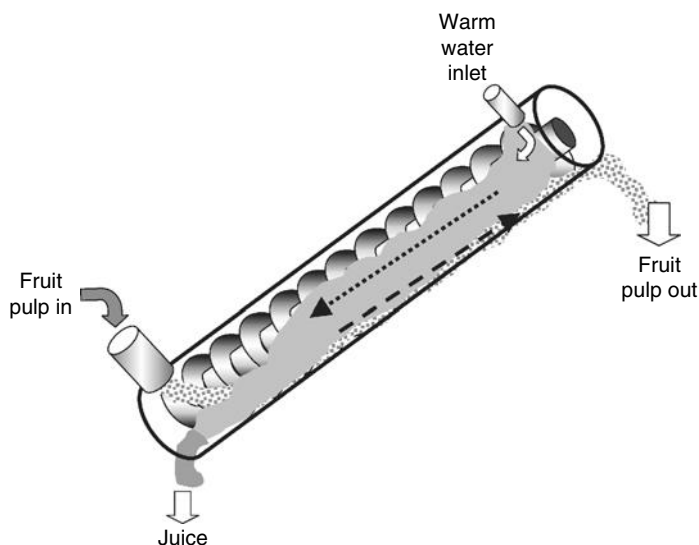


Figure 2.11. Sketch of fruit juice diffusion extraction process.

### 2.2.3. Clarification and Fining

The conventional route to concentration is to strip aroma, then depectinize juice with enzymes, centrifuge to remove heavy sediments and filter through pressure precoat filters and polish filters (Figure 2.2a).

The juice is then usually concentrated through a multistage vacuum concentrator. This process involves a slight decrease in concentration of juice during the stripping step (usually up to 10% volume is removed). Stripping usually precedes depectinization, as pectin methyl esterase releases significant quantities of methanol, which spoils the essence. The use of enzymes for clarification is described later in this chapter. When a more concentrated juice is clarified ( $\cong 20^\circ$  Brix) the volume to handle is reduced practically in a half. However, viscosity increased with concentration, which may slow flocculation and filtration. If a cloudy product is required, the juice is pasteurized immediately after pressing to denature any residual enzymes. Centrifugation then removes large pieces of debris, leaving most of the small particles in suspension.

#### 2.2.3.1. Partial Concentrates

Fruit juices, both clarified or opalescent, may be concentrated up to 4 fold ( $\cong 50^\circ$  Brix) with natural pectin gelling with little effort. At this point in the concentration process little heat damage is detected. This concentrate can be canned and frozen. For clear juice these suspended particles have to be removed (McLellan, 1996). It may seem simple merely to filter them out, but unfortunately some soluble pectin remains in the juice, making it too viscous to filter quickly. A dose of commercial enzyme is the accepted way of removing unwanted pectin.

*Depectinization* has two effects: it degrades the viscous soluble pectins and it also causes the aggregation of cloudy particles. Pectin forms a protective coat around proteins in suspension. In an acidic environment (apple juice typically has a pH of 3.5) pectin molecules carry a negative charge. This causes them to repel one another. Pectinolytic enzymes degrade pectin and expose part of the positively charged protein beneath. As the electrostatic repulsion between cloudy particles is reduced, they clump together. These larger particles will eventually settle, but to improve the process flocculating agents (fining) such as gelatin, tannin, or bentonite (a type of clay) can be added. Some fining agents adsorb the enzyme onto their surface, so it is important not to add them before the enzyme has done its job.

Fining agents (Table 2.5) work either by sticking to particles, thereby making them heavy enough to sink; or by using charged ions to cause particles to stick to each other, thereby making them settle to the bottom. Although this method of conventional clarification was widely used in the clarified juice industry, this technology has been practically replaced by mechanical processes such as ultrafiltration and centrifugal decanters.

Yeasts and other microbes, which may have contaminated the juice, may also be precipitated by fining. What is left is a transparent, but by no means, clear juice. A second centrifugation and a subsequent filtration are needed to produce the clear juice that many consumers prefer.

Another potential contributor to the haziness of juice is starch. This is particularly so if unripe apples have been used. Unripe apples may contain up to 15% starch. Although the first centrifugation—before the juice reaches the clarification tank—removes most of the starch, about 5% usually remains. This can be broken down using an amylase (amylglucosidase) active at the pH of apple juice, added at the same time as the pectinase.

**Table 2.5.** Fruit juice clarification agents.

Name	Description
Sparkolloid	Sparkolloid is a natural albuminous protein extracted from kelp and sold as a very fine powder
Gelatin	It is in general a mixture of gelatins and silicon dioxide, with animal collagen being the active ingredient
Kieselsoil	Kieselsoil is a liquid in which small silica particles have been suspended. It is usually used in tandem with gelatin. The dosage is 1 ml/g of gelatins. This fining aids in pulling proteins out of suspension
Bentonite	Bentonite is sold as a powder and as coarse granules. It is refined clay. A better way is to add the same amount to a liter of hot water, stir well, and let stand for 36–48 h. In this time the clay swells and becomes almost a gelatin
Isinglass	Produced from sturgeon swim bladders, isinglass is sold either as a fine white powder or as dry hard fragments. It is a protein extracted from the bladders of these fish. This product is also available as a prepared liquid called “super-clear”
Filters or Polishers	With a fine porosity pad, filters are very effective in removing particles (yeast cells, proteins, etc.)
Pectic Enzymes	Almost all fruits contain pectin, some more than others. When added as directed, it eliminates pectin haze. There is no other way to prevent this condition, and if it is in a juice, the haze will never clear on its own

For juice processing both depectinization and destarching are essential. This is because most apple juice is concentrated by evaporating up to 75% of the water content before storage. This makes the juice easier to transport and store, and the concentrate’s high sugar content acts as a natural preservative.

Unfortunately, heat treatment also drives off the juice’s pleasant aroma, so it is necessary to gently heat the juice and collect the volatile smell and flavor compounds, so that they can be put back again when the juice is reconstituted. Heating can cause residual pectin or starch in the juice to gel or form a haze, hence the necessity of enzyme treatment. Increased haze formation occurs when fining with gelatin and bentonite is not performed.

Optimization of fining and ultrafiltration steps can help retard or prevent postbottling haze development.

#### **2.2.4. Use of Enzymes in the Fruit Industry**

Commercial pectic enzymes (pectinases) and other enzymes are now an integral part of fruit juice technology (Grampp, 1976). They are used to help extract, clarify, and modify juices from many fruits, including berries, stone and citrus fruits, grapes, apples, pears, and even vegetables. When a cloudy juice or nectar is preferred (for example, with oranges, pineapples, or apricots) there is no need to clarify the liquid, and enzymes are used to enhance extraction or perform other modifications. The available commercial pectinase preparations used in fruit processing generally contain a mixture of pectinesterase (PE), polygalacturonase (PG), and pectinlyase (PL) enzymes (Dietrich et al., 1991). Enzymatic juice extraction from apples was introduced 25 years ago, and now some 3–5 million tons of apples are processed into juice annually throughout the world. The methods employed for apple juice are generally the same as those for other fruits (Table 2.6).

As previously mentioned, after fruits like apples have been washed and sorted, they are crushed in a mill. Peels and cores from apple slice or sauce production may also be used



**Table 2.6.** Application of pectolytic enzymes to fruit and vegetable processing.

Enzymatic process	Examples of application
Clarification of fruit juices	Apple juice, depectinized juices can also be concentrated without gelling and developing turbidity
Enzyme treatment of pulp	Soft fruits, red grapes, citrus, and apples; for better release of juice (and colored material); enzyme treatment of pulp of olives, palm fruit, and coconut flesh to increase oil yield
Maceration of fruit and vegetables (disintegration by cell separation)	Used to obtain nectar bases and in baby foods
Liquefaction of fruit and vegetables	Used to obtain products with increased soluble solids' content (pectinases and cellulases combined)
Special applications to citrus fruits	Used for the preparation of clouding agents from citrus peel, cleaning of peels before use in candy and marmalade production, recovery of oil from citrus peel, depectinization of citrus pulp wash

(Source: Rombouts and Pilnik, 1978).

together with whole apples. Although pectinases are often added at this stage, better results are achieved if the apple pulp is first stirred in a holding tank for 15–20 minutes so that enzyme inhibitors (polyphenols) are oxidized (by naturally occurring polyphenols oxidized in the fruit). The pulp is then heated to an appropriate temperature before enzyme treatment. For apples 30°C is the optimal temperature, whereas stone fruits and berries generally require higher temperatures—around 50°C. This compares with 60–65°C required if pectinase is not used (here the juice is liberated by plasmolysis of the plant cells).

Prepress treatment with pectinases takes anything from 15 min to 2 h depending upon the exact nature of the enzyme and how much is used, the reaction temperature, and the variety of apple chosen. Some varieties such as Golden Delicious are notoriously difficult to breakdown. During incubation the pectinase degrades soluble pectin in the pulp, making the juice flow more freely. The enzyme also helps to breakdown insoluble pectin, which impede juice extraction.

Enzyme treatment is considered to be complete once the viscosity of the juice has returned to its original level or less. It is important that the pulp is not broken down too much as it would then be difficult to press.

Pressing is done using the previously described equipment. Juice yields can be increased by up to 20%, depending upon the age and variety of fruit used and whether preoxidation was employed. Enzyme treatment is particularly effective with mature apples and those from cold storage.

#### 2.2.4.1. Other Enzymes in Juice Production

- *Cellulases*: The addition of cellulases during extraction at 50°C improves the release of color compounds from the skins of fruit. This is particularly useful for treating blackcurrants and red grapes. Increasingly cellulases are being used at the time of the initial pectinase addition to totally liquefy the plant tissue. This makes it possible to filter juice straight from the pulp without any need for pressing.
- *Arabanase*: The polysaccharide araban (a polymer of the pentose arabinose) may appear as a haze in fruit juice a few weeks after it has been concentrated. Although commercial pectinase preparations often contain arabanase, certain fruits (like pears)

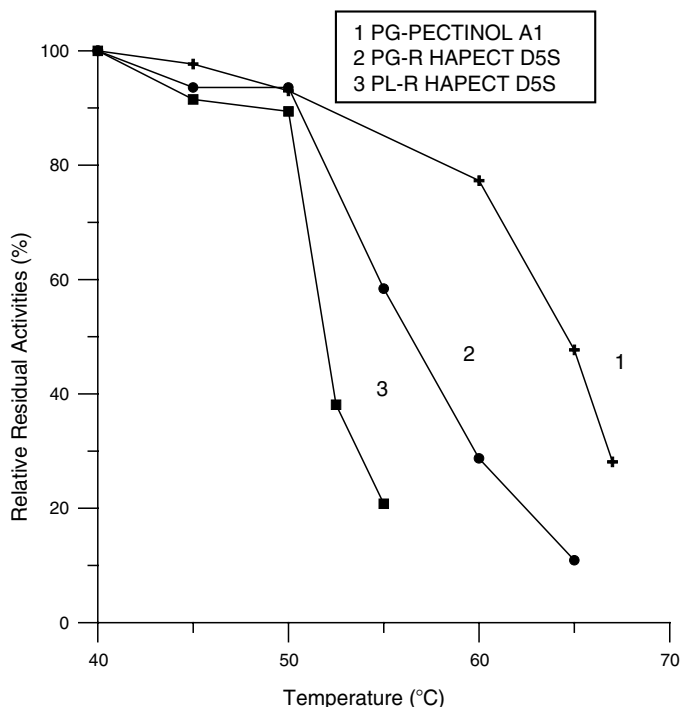
are rich in araban and may require the addition of extra arabanase to the clarification tank.

- *Glucose oxidase* (from the fungi *Aspergillus niger* or *Penicillium* spp.): Catalyzes the breakdown of glucose to produce gluconic acid and hydrogen peroxide. This reaction utilizes molecular oxygen. Glucose oxidase (coupled with catalase to remove the hydrogen peroxide) is therefore used to remove the oxygen from the head space above bottled drinks, thereby reducing the nonenzymatic browning due to oxidation, which might otherwise occur.

#### 2.2.4.2. Pectinase Activity Determination

Complete pectin breakdown in apple juices can be ensured only if all the three types of pectinolytic enzymes (PG, PE, PL) are present in the correct proportion. Apple juice processor in general lacks reliable methods for checking the different enzyme activities. Application and success of a pectinase product also depend on the substrate where they act. The problems in evaluation of pectinolytic activities are caused by the difficulty in standardizing fruit substrate. Acidity, pH, and the presence of inhibitors or promoters of the enzymatic reaction depend upon the variety of apple processed.

Figure 2.12 shows the residual polygalacturonase activity of two commercial enzymes after 30 min of heating at different temperatures. They start to become inactivated at temperatures higher than 50°C, which is a very well defined breaking point, if the enzyme



**Figure 2.12.** Enzymatic residual activities after thermal treatment 30 min at different temperatures) of enzyme solutions in 0.1 M citrate/0.2 M phosphate buffers at optimum pH). Reprinted from Food Chem. 31(½), Ceci, L. and Lozano, J.E. Determination of enzymatic activities of commercial pectinases, 237–241. (copyright) 2003, with permission from Elsevier.

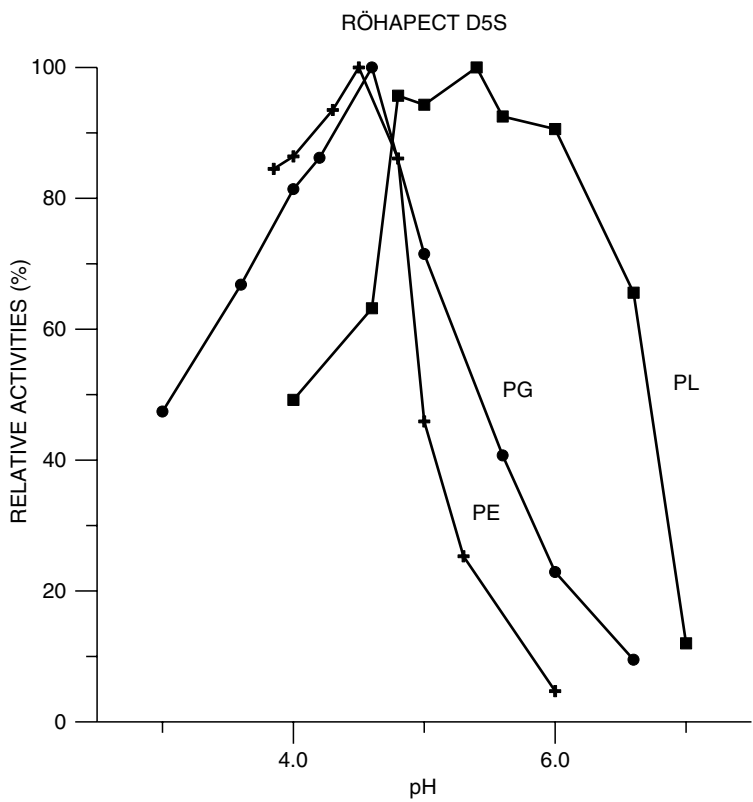
were to rapidly loses its activity. The rate of PG activity decrease could be divided into two periods (Fig. 2.12). The first period was characterized as a thermolabile fraction. The second can be defined as the thermoresistant fraction of the enzyme. Sakai et al. (1993) and Liu and Luh (1978) reported that the optimal temperature for PG activity was in the range 30–50°C.

Both authors indicated that for temperatures greater than 50°C inactivation was notable after a short period of heating. Moreover, the optimal temperature is also a function of the type of substrate to be treated (Ben-Shalom et al., 1986).

Inactivation curve of lyase activity (PL) is also shown in Fig. 2.12. As in the case of PG activities, 50°C can be easily identified as the breaking point where PL rapidly inactivates. Alkorta et al. (1996) found that PL from *Penicillium italicum* was active after 1 h at 50°C but resulted in complete inactivation for the same period at 60°C. The commercial enzymes proved to be more heat tolerant than purified fractions (Liu and Luh 1978). This phenomenon was attributable to the heat protective action of impurities.

#### 2.2.4.3. pH Dependence on the Pectic Enzymes Activities

Figure 2.13 shows the behavior of PG and PE activities of RHD5 enzyme versus pH. The resulting optimum pH was approximately 4.6. However, the curve for lyase activity as a



**Figure 2.13.** Effects of pH on the enzymatic activities of Röhaupt D5S (Ceci and Lozano, 1998). Reprinted from Food Chem. 31(½), Ceci, L. and Lozano, J.E. Determination of enzymatic activities of commercial pectinases, 237–241. (copyright) 2003, with permission from Elsevier.

function of pH was much broader, and it was difficult to identify a single optimal value. In this case, an optimal range of pH 5–6 may be defined.

As a result, as much as 40% of PG and PE inactivation can be expected during the enzymatic clarification of the relatively acidic Granny Smith juice. It was found that a shift in the optimal pH toward the acid zone (Spagna et al., 1993) or a broadening of the optimal activities' range (Ates and Pekyardimci, 1995) can be obtained after enzyme immobilization on appropriate supports.

In general pectinolytic enzymes (PG and PE) show a rapid decrease in activity at about pH 5 and become practically inactivated near neutrality. However, this problem becomes irrelevant because pH values of fruit juices are lower than pH 5. It was found that a shift in the optimal pH toward the acid zone (Spagna et al., 1993) or a broadening of the optimal activities' range (Ates and Pekyardimci, 1995) can be obtained after enzyme immobilization on appropriate supports

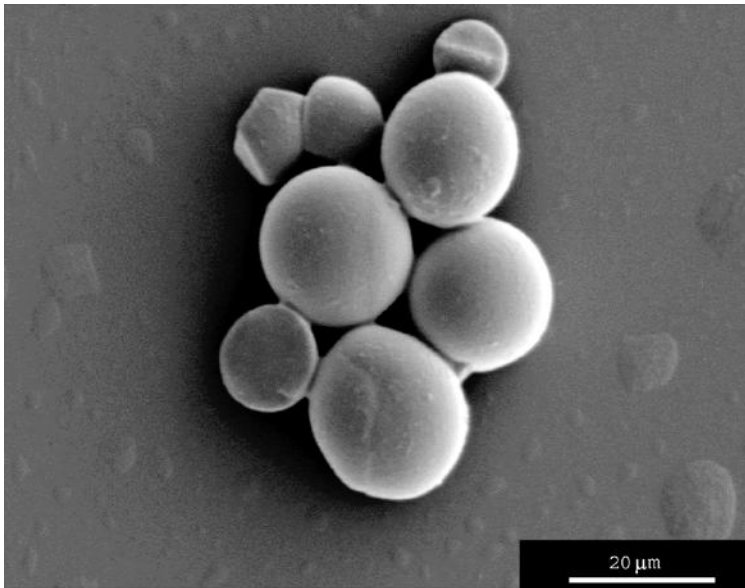
As fruit juice clarification is usually done at 45–50°C, special care must be taken to avoid excessive inactivation when lyase activity is considered important. It is known (Dietrich et al., 1991) that commercial enzyme preparations cause a certain degree of side activities other than those required (Ceci and Lozano, 1998).

#### 2.2.4.4. *Enzymatic Hydrolysis of Starch in Fruit Juices*

Starch can be a problem for juice processors. Polymeric carbohydrates like starch and arabans can be difficult to filter and cause postprocess cloudiness. In the case of a positive starch test, the following problems may occur: slow filtration, membrane fouling, gelling after concentration, and postconcentration haze. Apple juice is one of the juices that can contain considerable amounts of starch, particularly at the beginning of the season. Unripe apples contain as much as 15% starch (Reed, 1975). As the apple matures on the tree, the starch hydrolyzes into sugars. The starch content of apple juice may be high in years when there were relatively low temperatures during the growing season. Besides the generalized application of commercial amylase enzymes in the juice industry, there is a lack of information on apple starch characteristics and extent of gelatinization during juice pasteurization.

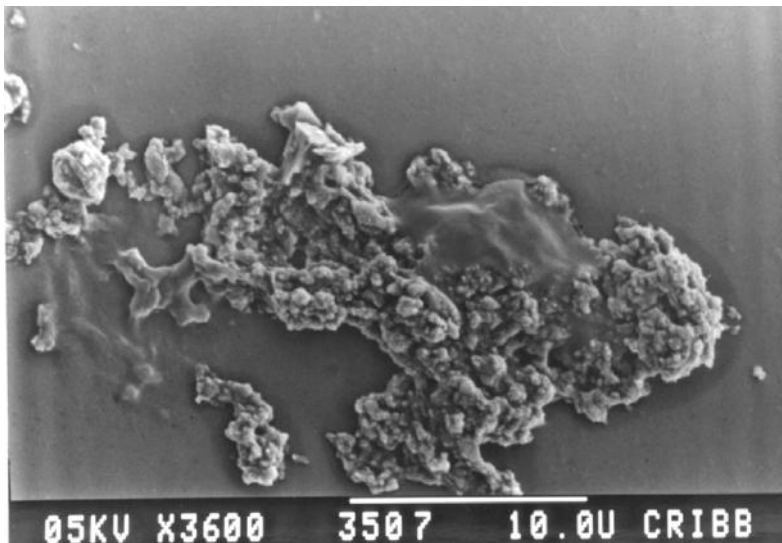
Starch must be degraded by adding starch-splitting enzymes, together with the pectinase during depectinization of the juice. First starch must be gelatinized, by heating the juice to 77°C. When an aqueous suspension of starch is heated the hydrogen bonds weaken; water is absorbed; and the starch granules swell, rupture, and gelatinize (Zobel, 1984). The juice must then be cooled to <50°C to avoid enzyme inactivation. Starch is generally insoluble in water at room temperature. Because of this, it is stored in cells as small granules.

Starch granules (Fig. 2.14) are quite resistant to penetration by both water and hydrolytic enzymes due to the formation of hydrogen bonds within the same molecule and with other neighboring molecules. When the starch granule is not broken down completely, a short-chained dextrin is left. This can lead to a condition known as retrograding. When starch retrogrades, the short-chained dextrin recrystallizes into a form that is no longer susceptible to enzyme attack, regardless of heating. Figure 2.15 shows a SEM photomicrograph of haze sediment obtained from a pasteurized apple juice sample.



**Figure 2.14.** Scanning electron photomicrograph of an isolated apple starch granule (5 kV  $\times$  4,400).

The scanning electron micrograph shows how the apple starch granules collapsed after heat treatment, while dispersion of gel-like starch fragments among the other components of turbidity (pectin, cellular wall, etc.) can be observed. Similar behavior was found when wheat starch was gelatinized by heat in excess water (Lineback and Wongsrikasen, 1980).



**Figure 2.15.** Scanning electron photomicrograph of cloudiness precipitated from a pasteurized (5 min at  $90^{\circ} \pm 1^{\circ} \text{C}$ ) apple juice (5 kV  $\times$  3,600).

### 2.2.5. Filtration

Filtration is also a mechanical process designed for clarification by removing insoluble solids from a high-value liquid food, by the passage of most of the fluid through a porous barrier, which retains most of the solid particulates contained in the food. Filtration is performed using a filter medium, which can be a screen, cloth, paper, or bed of solids. Filter acts as a barrier that lets the liquid pass while most of the solids are retained. The liquid that passes through the filter medium is called the filtrate. There are several filtration methods and filters (Lozano, 2003) including:

- *Driving force*: The filtrate is induced to flow through the filter medium by: (a) hydrostatic head (gravity), (b) pressure (upstream filter medium), (c) vacuum (downstream filter medium), and (d) centrifugal force across the medium.
- *Filtration mechanism*: (a) cake filtration (solids are retained at the surface of a filter medium and pile upon one another). (b) depth or clarifying filtration (solids are trapped within the pores or body of the filter medium).
- *Operating cycle*: (a) Intermittent (batch) and (b) Continuous rate.

These methods of classification are not mutually exclusive. Thus filters usually are divided first into the two groups of cake and clarifying equipment, then into groups of machines using the same kind of driving force, then further into batch and continuous classes. Some filtering devices usually employed in the food industry are described here.

#### 2.2.5.1. Pressure Filters

The main advantages of pressure filtration compared to other filtering methods are: Cakes are obtained with very low moisture content, clean filtrates may be produced by recirculating the filtrate or by precoating, and the solution can be polished (finished) to a high degree of clarity. Among disadvantages it must be noted that cloth washing is difficult, and if precoat is required, the operator cannot see the forming cake and is unable to carry out an inspection while the filter is in operation, and the internals are difficult to clean, which can be a problem with food-grade applications.

With the exception of the rotary drum pressure filter, this type of filters has a semicontinuous machine in which wash and cake discharge are performed at the end of the filtration cycle. Since the operation is in batches, intermediary tanks are required. The collection of filtrate depends on the operating mode of the filter, which can be at constant flow rate, constant pressure, or both, with pressure rising and flow rate reducing during filtration. The filtration rate is mainly influenced by the properties of food (particle size and distribution, presence of gelatinous solids like pectin, liquid viscosity, etc.) Although continuous pressure filters are available, they are mechanically complex and expensive, so they are not common in the food industry.

#### 2.2.5.2. Filter Aid and Precoating

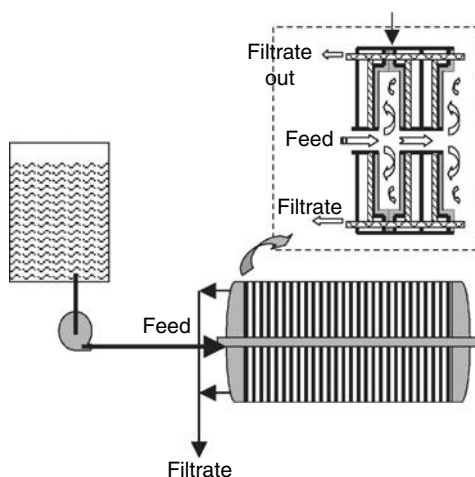
Filter aid and precoating are often used in pressing and in connection with pressure filtration. Filter aid is used when the pulp or turbid liquid food is low in solids' content with fine and muddy particles that are difficult to filter. To enhance filtration coarse solids with large surface area that capture and trap the slow-filtering particles from the suspension in its interstices and produces a porous cake matrix are used.

On the other hand, precoat is the formation of a defined thick medium of a known permeability on the filter plates. Precoating prior to filtration serves when the particles that are to be separated are gelatinous and sticky, forming a barrier that avoids cloth blinding. The most common filter aids and precoating materials employed in the food industry are: diatomaceous earth (siliceous skeletal remains of aquatic unicellular plants), perlite (glassy crushed and heat-expanded volcanic rock), cellulose (fibrous light weight and ashless paper), and special groundwood.

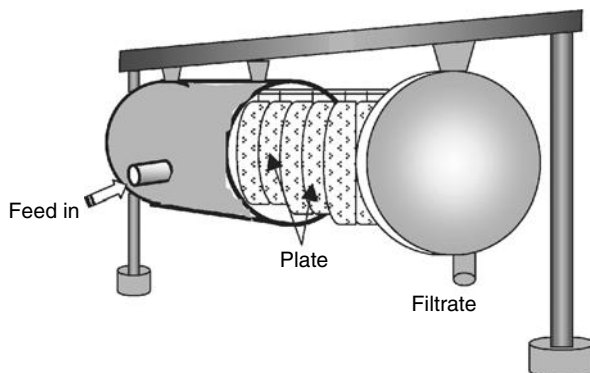
### 2.2.5.3. Types of Pressure Filters

Pressure filters usually found in the food industry are:

- *Filterpress*, also called *Plate and Frame*, consists of a head and a follower that contain in between a pack of vertical rectangular plates that are supported by side or overhead beams (Fig. 2.16). The head serves as a fixed end to which the feed and filtrate pipes are connected, while the follower moves along the beams and presses the plates together during the filtration cycle by a hydraulic or mechanical mechanism. Each plate is covered with filter cloth on both sides and, once pressed together, they form a series of chambers that depend on the number of plates. The plates have generally a centered feed port that passes through the entire length of the filter press, so that all chambers of the plate pack are connected together.
- *Vertical and horizontal pressure leaf filters* consist of a vessel that is fitted with a stack of vertical (Fig. 2.17), or horizontal leaves that serve as filter elements. The leaf is constructed with ribs on both sides to allow free flow of filtrate toward the neck and is covered with coarse mesh screens that support the finer woven metal screens or filter cloth that retain the cake. The space between the leaves varies from 30 to 100 mm depending on the cake formation properties and the ability of the vacuum to hold a thick and heavy cake to the leaf surface. The filters can be used for polishing fruit juices



**Figure 2.16.** Filterpress operation sketch and details of filtering plate. Reprinted from the Encyclopedia of Food Science and Nutrition, Lozano J.E., Separation and Clarification, pp. 5187–5196. (copyright) 2003, with permission from Elsevier.



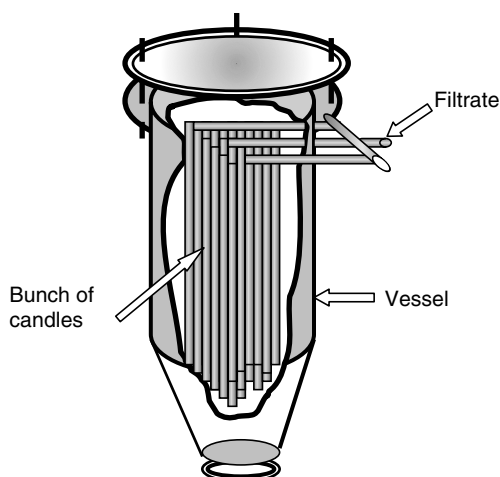
**Figure 2.17.** Horizontal plate filter. Reprinted from the Encyclopedia of Food Science and Nutrition, Lozano J.E., Separation and Clarification, pp. 5187–5196. (copyright) 2003, with permission from Elsevier.

with very low solids or for cake filtration with a solids' concentration of  $<20\text{--}25\%$ . The cloth mesh screens that cover the leaves can be more easily accessed on horizontal tanks than on vertical tanks.

- *Candle filters* are used in applications that require efficient low moisture cake filtration or high degree of polishing (finishing). Candle filters can contain more than 250 filtering elements. They consist basically of three major components (Fig. 2.18):
  - (1) The vessel;
  - (2) The filtering elements (candles) and
  - (3) The cake discharge outlet.

The vessel configuration has a conical bottom for cake filtration and polishing, or has a dished bottom for slurry thickening, though it is scarcely used in the food industry.

The filtering element generally consists of the filtrate core and the filtering medium. The core helps in filtrate passage and supports the filter medium. The core is a bundle of



**Figure 2.18.** Candle filter sketch showing major components. Reprinted from the Encyclopedia of Food Science and Nutrition, Lozano J.E., Separation and Clarification, pp. 5187–5196. (copyright) 2003, with permission from Elsevier.



perforated stainless steel tubes. The filter medium can be a porous ceramic, woven mesh screen, sintered metal tube, or synthetic filter cloth. The advantages of a candle filter are the excellent cake discharge capacity and its mechanical simplicity.

#### 2.2.5.4. Vacuum Filters

Vacuum filters are simple and reliable machines widely used in the fruit industry. Among the different types of vacuum filter (drum, disk, horizontal belt, tilting pan, and table filters) drum filters are most commonly utilized in the food industry. The advantages and disadvantages of vacuum filtration compared to other separation methods are:

- *Advantages:* Continuous operation, very effective polishing (finishing) of solutions (on a precoat filter), and easy control of operating parameters such as cake thickness.
- *Disadvantages:* Higher residual moisture in the cake and difficulty in cleaning (as required mainly for food-grade applications).

Precoat filters are used when liquid foods (e.g., clarified apple juice) require a very high degree of clarity. To polish the solution the drum deck is precoat with an appropriate medium (See Filter aids and precoating in this chapter). A scraper blade also called “Doctor Blade,” moves slowly toward the drum and shaves off a thin layer of the separated solids and precoat material. This movement exposes continuously a fresh layer of the precoat surface, so that when the drum submerges into the tank it is ready to polish the solution. Precoat filters are used to recover juice from the sediments originating in clarification tanks. In precoat filters the entire drum deck is subjected to vacuum (Fig. 2.19).

#### 2.2.6. Membrane Filtration

Filtration of coarse particles down to several microns is done by conventional dead-end filtration, where all influent passes through a filter medium that removes contaminants to produce higher-quality clarified juices. Rough screens, sand filters, multimedia filters, bag filters, and cartridge filters are examples of filtration products that remove 0.1-micron size particles or larger. Once the medium becomes loaded, it can be backwashed as with multimedia filters, or discarded and replaced as with cartridge filters. The method of obtaining clean filtration medium is based on economic and disposal concerns. Particles retained by the filter in dead-end filtration build up with time as a cake layer, which results in increased resistance to filtration. This requires frequent cleaning or replacement of filters.

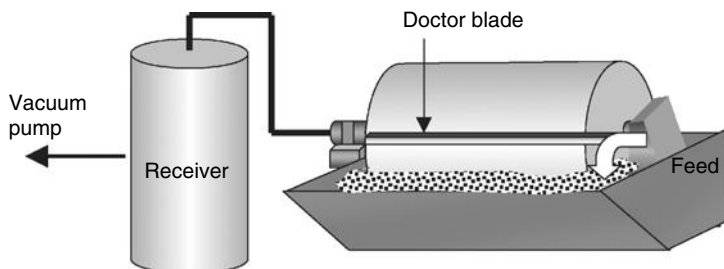
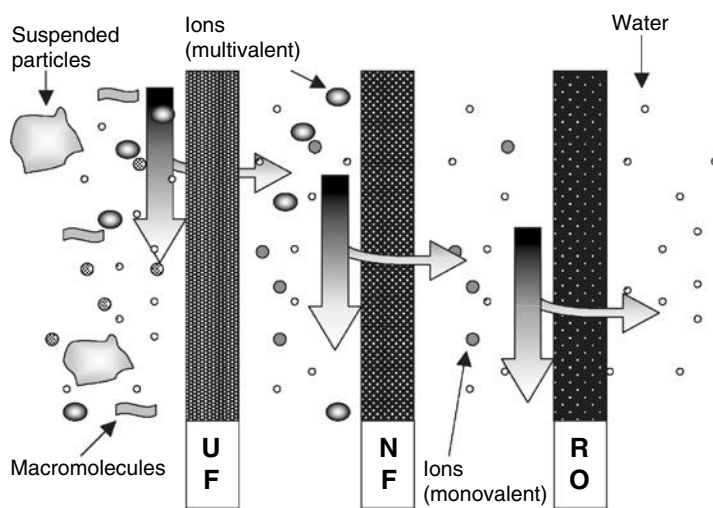


Figure 2.19. Vacuum drum precoat filter.

Commercially available coarse filtration devices are effective in separating particles down to about  $20\text{ }\mu\text{m}$ . On the other hand, membrane technology involves the separation of particles below this range, extending down to dissolved solutes that are as small as several Angstroms.

Membranes are manufactured with a wide variety of materials, including sintered metals, ceramics, and polymers (Zeman and Zydney, 1996). In order to reject substances smaller than  $0.1\text{ }\mu\text{m}$  using polymeric membrane is by far the most popular filtration method in the fruit processing industry. Millions of small pores per unit area of membrane allow water and low-molecular weight substances to pass through it while undesired substances are retained on the influent side. The problem is solved by operating polymeric membranes in the *crossflow* mode. In crossflow membrane filtration, two effluent streams are produced, the permeate and the concentrate. The permeate is the purified fluid that has passed through the semi-permeable membrane. The remaining fluid is the concentrate, which has become enriched with organics and salts that could not permeate the membrane. By doing so, rejected contaminants are continuously carried away from the membrane surface, thereby minimizing contaminant build up, leaving it free to reject incoming material and allow free flow of purified liquid. The size of the polymer's pores categorizes the membrane into one of the following groups: reverse osmosis, nanofiltration, ultrafiltration, and microfiltration. Reverse osmosis (RO) membranes have the smallest pore size ranging from 5 to 15 angstroms, nanofiltration (NF) covers separations in the 15–30 angstrom size, ultrafiltration (UF) removes organics in the  $0.002\text{--}0.2\text{ }\mu\text{m}$  range, while microfiltration (MF) effects separation typically in the range of  $0.1\text{--}10\text{ }\mu\text{m}$ .

Figure 2.20 schematically shows the filtering capacity of these “crossflow” membrane systems. RO is both a mechanical and chemical filtration procedure by which the membrane's surface sieves organic substance and actually repels ions. The dielectric repulsion of ions from the membrane is influenced by the ion's charge density. Unlike RO membranes that have salt retentions of 80–99%, NF membranes reject 30–60% salts. UF and MF membranes have even larger pores and therefore pass most of the salts. Although membrane cleaning is periodically

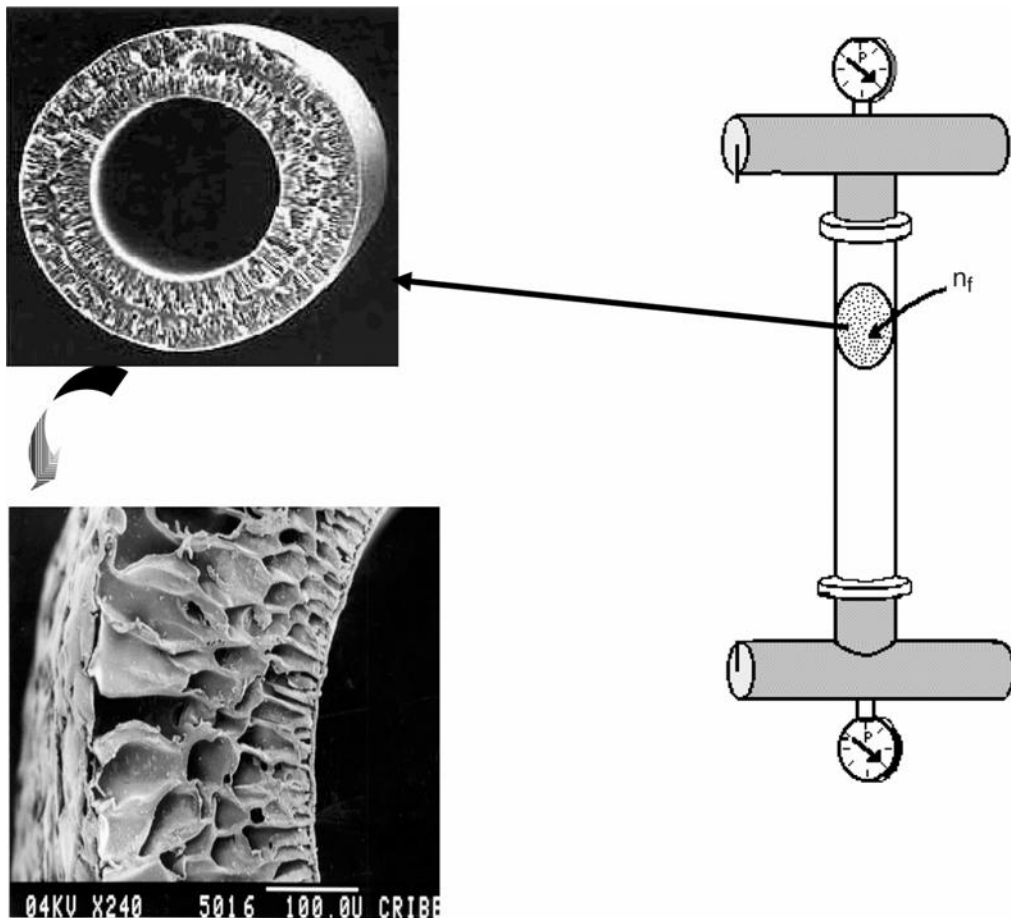


**Figure 2.20.** Crossflow-type membrane classification by “rejection” capacity.

required, the self-cleaning nature of crossflow filtration lengthens membrane life enough to make it economically attractive.

The manufacturing processes result in a number of different membrane structures such as microporous, asymmetric, composite, etc. Membranes are assembled as modules that are easily integrated into systems containing hydraulic components. The module allows to accommodate large filtration areas in a small volume and resist the pressures required in filtration. Tubular, hollow fiber, spiral, and flat plate are the common modules (Cheryan, 1986).

- *Tubular module* consists of tubular membranes held inside individual perforated support tubes, assembled onto common headers and permeate into the container to form a module. When several channels are formed in a porous block of material, the tubular system is called “monolithic.”
- *Hollow fiber module* consists of bundles of hollow fibers (0.5–3 mm internal diameter) sealed into plastic headers and assembled in permeate casings (Fig. 2.21). Hollow fibers



**Figure 2.21.** Hollow fiber membrane configuration. (a) Manifold with HF cartridge; (b) SEM micrograph of a single fiber (internal diameter 1 mm); (c) SEM magnification of a single fiber, showing filtration surface and support.

are used in low-pressure applications only. These can accommodate moderate levels of suspended particles.

- *Spiral modules* are made by placing a woven plastic mesh, which acts as the permeate channel between two membrane layers and seals three sides. The fourth side of this sandwich is attached to the permeate tube. Another plastic mesh that acts as the feed channel is laid over it and the assembly is wrapped around the central permeate tube.
- *Flat-plate modules* use multiple flat sheet membranes in a sandwich arrangement consisting of a support plate, membrane, and channel separator. The membranes are sealed to the plates using gaskets and hydraulically clamped to form a tight fit. Several of these membranes are stacked together and clamped to form a complete module. The main advantages of the flat-plate module design are that they have high membrane-packing densities and low hold-up volumes. This is due to the small channel height of the flat-plate modules. The main application for the flat-plate module is in recovering biological products. The advantages and disadvantages of the different UF configurations (Cheryan, 1986) are listed in Table 2.7.

Over time, the physical backwash will not remove some membrane fouling. Most membrane systems allow the feed pressure to gradually increase over time to around 30 psi and then perform a clean-in-place (CIP). CIP frequency might vary from around 10 days to several months. Another approach CIP practice is to use a chemically enhanced backwash (CEB), where on a frequent basis (typically every 1–14 days), chemicals are injected with the backwash water to clean the membrane and maintain system performance at low pressure without going offline for a CIP.

The application of ultrafiltration (UF) as an alternative to conventional processes for clarification of apple juice was clearly demonstrated (Heatherbell et al., 1977; Short, 1983; Wu et al., 1990). However, the acceptance of UF in the fruit processing industry is not yet complete, because there are problems with the operation and fouling of membranes. During UF two fluid streams are generated: the ultrafiltered solids' free juice (*permeate*), and the *retentate* with variable content of insoluble solids, which, in the case of apple juice, are mainly remains of cellular walls and pectin.

Permeate flux ( $J$ ) results from the difference between a convective flux from the bulk of the juice to the membrane and a counterdiffusive flux or outflow by which the solute is transferred back into the bulk of the fluid. The value of  $J$  is strongly dependent on hydrodynamical conditions, membrane properties, and the operating parameters. The main driving force of UF is the transmembrane pressure ( $\Delta P_{TM}$ ), which in the case of hollow fiber ultrafiltration systems (HFUF) can be defined as:

$$\Delta P_{TM} = \frac{(P_i + P_o)}{2} - P_{ext} \quad (2.1)$$

**Table 2.7.** Advantages and disadvantages of the different UF configurations.

	UF MEMBRANE CONFIGURATION			
	Flat/press	Spiral	Tubular	Hollow fibers
Pack density ( $\text{m}^2/\text{m}^3$ )	300–500	200–800	30–200	500–9000
Fouling resistance	Good	Moderate	Very good	Poor
Cleaning facility	Good	Fair	Very good	Poor
Relative cost	High	Low	High	Low

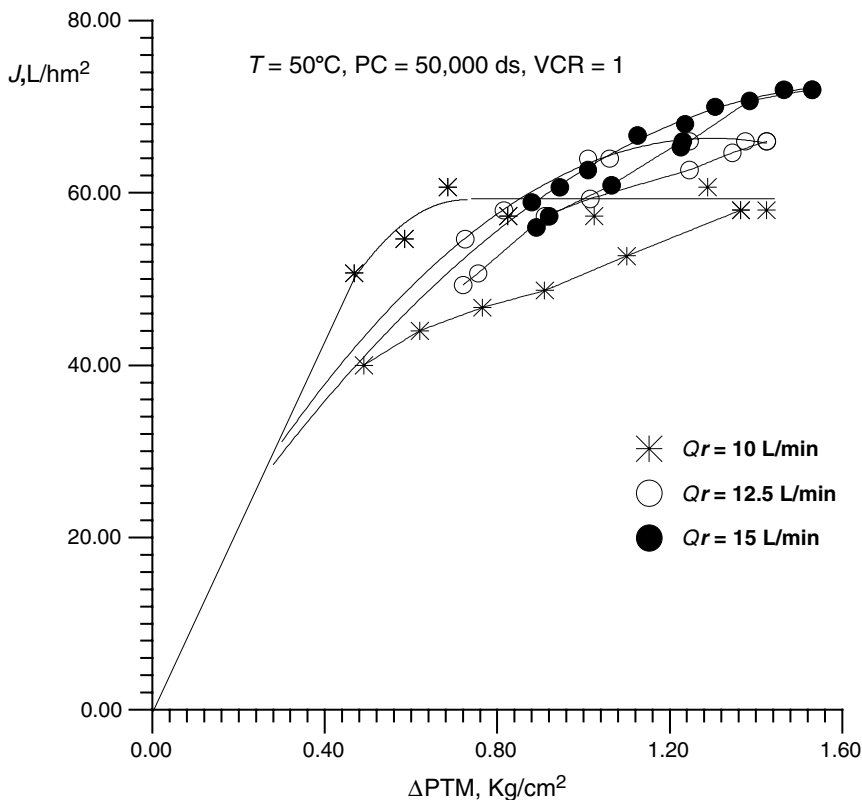
Source: Cheryan, 1986; Zeman and Zydney, 1996.

where  $P_i$  is the pressure at the inlet of the fiber,  $P_o$  the outlet pressure, and  $P_{ext}$  the pressure on the permeate side. In practice, the  $J$  values obtained with apple juice are much less than those obtained with water only. This phenomenon is attributable to various causes, including resistance of gel layer, concentration in polarization boundary layer (defined as a localized increase in concentration of rejected solutes at the membrane surface due to convective transport of solutes (Porter, 1972)), and plugging of pores due to fouling, where some of these phenomena are reversible and disappear after cleaning of the UF membranes while others are definitively irreversible.

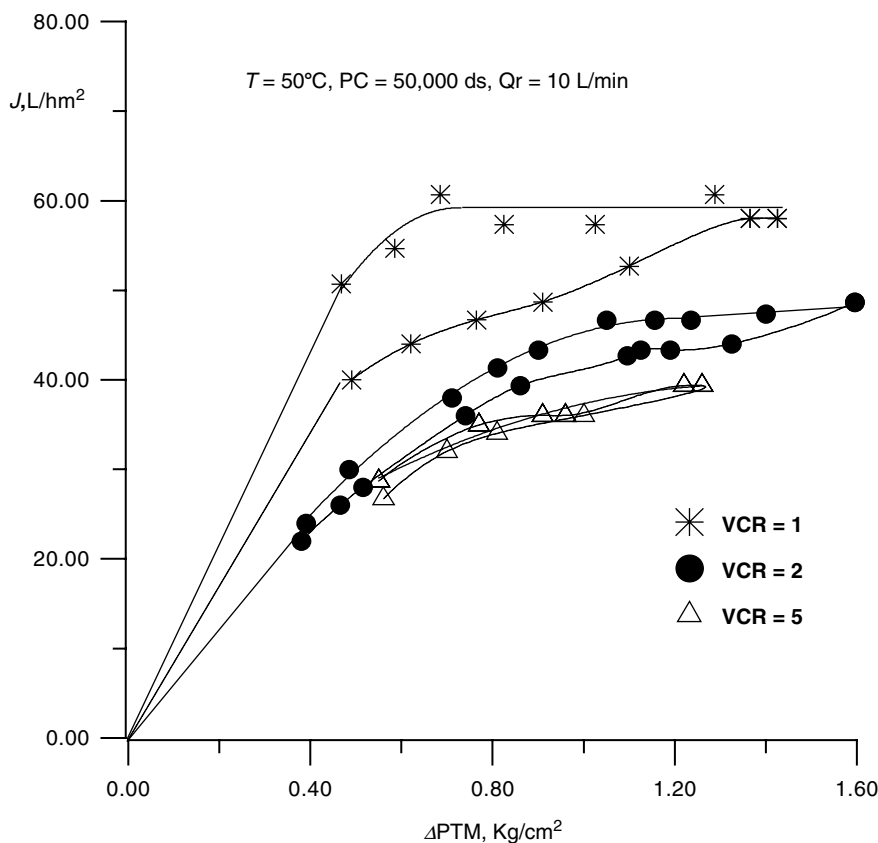
### 2.2.6.1. Stationary Permeate Flux

It is well known (Iritani et al., 1991) that the transmembrane pressure-permeate flux characteristic for ultrafiltration shows a linear dependence of  $J$  with  $\Delta PTM$  at lower values of pressure (1st region), while the permeate flux approaches a limiting value ( $J_{lim}$ ) independent of further increase in pressure at higher pressures (2nd region). The last situation was assumed to be controlled by mass transfer.

Figures 2.22 and 2.23 show the variation of  $J$  with  $\Delta PTM$  as a function of VCR or volume concentration ratio (defined as the initial volume divided by retentate volume at any time), which is a measurement of the retentate concentration, and recirculating flow rate,  $Qr$ , respectively (Constenla and Lozano, 1996). Pressure independence (2nd region) was observed to occur at a higher pressure at higher  $Qr$ . The point at which the pressure independence is evident is called optimum transmembrane pressure ( $\Delta PTM_o$ ).



**Figure 2.22.** Effect of  $\Delta PTM$  and  $Qr$  on  $J$  at  $50^\circ\text{C}$ . (Constenla and Lozano, 1996) with permission.



**Figure 2.23.** Effect of  $\Delta\text{PTM}$  and VCR on  $J$  at  $50^{\circ}\text{C}$  (Constenla and Lozano, 1996). Reprinted from Lebensm. Wiss. u. Technol. 27: 7–14, Constenla, D.T. Lozano, J.E., Predicting stationary permeate flux in the ultrafiltration of apple juice. (copyright) 1996, with permission from Elsevier.

The reduction of  $J_{lim}$  with  $Q_r$  may be associated with a reduction in the boundary layer due to an increase in the turbulence. On the other hand, the optimal  $\Delta\text{PTM}$  values were practically independent of VCR at  $Q_r > 10$  L/min. A hysteresis effect in the permeate flux, attributable to the consolidation of the gel layer (Omosaiye et al., 1978), has been observed. The area enclosed by the hysteresis loop was greater at lower  $Q_r$  and VCR values. Traditionally, correlations of  $J$  with  $\Delta\text{PTM}$  and VCR were determined by parameter fitting of the experimental data. Since the polynomial functions have no physical basis, a large number of experimental data are needed for determination of  $J$ . Therefore other theoretical and semiempirical approaches should be considered (Constenla and Lozano, 1986).

#### 2.2.6.2. Permeate Flux as a Function of Time

Several models are proposed in the literature for representing  $J$ , most of them being semi-empirical and practical equations (Table 2.8). Membrane fouling mechanisms may be studied through the classical laws of filtration under constant pressure (Table 2.9). During UF process (Iritani et al., 1991)  $J$  behaves as in cake filtration only at the very beginning, attributable to the formation of the gel layer with minor counterdiffusion flux.

**Table 2.8.** Some equations representing permeate flux as a function of time.

No.	Permeate flux equations
(i)	$J = J_0 \exp(-Bt)$
(ii)	$J = J_F + (J_0 - J_F) \exp(-At)$
(iii)	$J = (J_0^{-2} + 2Kt)^{-1/2}$
(iv)	$J = J_0 - B \ln(VCR)$
(v)	$J - J_F = (J_0 - J_F)(1 - \exp(-(t/B)))$

*Subindex:* 0, 1, *F* are zero, initial, and final time, respectively;  $V_p$  = permeate volume; *A*, *B*, and *K* are constants.

*Sources:* Heatherbell et al. (1977), Probstein et al. (1979), Mietton-Peuchot et al. (1984), Koltuniewicz (1992), Constenla and Lozano (1996).

As previously indicated, pectin and other large solutes like starch, normally found when unripen apples are processed, tend to form a fairly viscous and gelatinous-type layer on the “skin” of the asymmetric fiber. Flux decline, due to this phenomenon, can be reduced by increasing flow velocity on the membrane

Traditionally correlations of *J* with ΔPTM and VCR have been determined by parameter fitting of the experimental data. It was found that the following exponential equation, proposed in the SRT model (Constenla and Lozano, 1996), fitted appropriately:

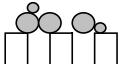
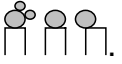

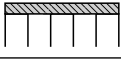
$$J = J_F + (J_0 - J_F) \exp(-At) \tag{2.2}$$

$J_0$ ,  $J_F$ , and *A* values can be obtained at different *Qr* and constant values of VCR and ΔPTM. An increase in *Qr* significantly increases the permeate flux. This behavior was reflected as an extensive increase in the parameter *A*.

### 2.2.6.3. Influence of VCR on the Permeate Flux

Constenla and Lozano (1996) found that in the case of pseudoplastic fluids, as fruit juice retentates, different operative conditions restrain the VCR up to a maximum of 14. The permeate *flux* becomes independent of the solute rejection, characteristic of the hollow fibers

**Table 2.9.** Classic filtration models (pseudoplastic fluids).

Mechanism	Scheme	Representative equations
(1) Total pore blocking		$J_0 - J = K_1 V_p \ln(J/J_0) = -K_1$
(2) Partial pore blocking		$\ln(J/J_0) = -K_2 V_{p1}/J - 1/J_0 = K_2 t$
(3) Blocking Progressive pore		$J = J_0(1 - K_3 V_p/2)(3n + 1)/2nJ$ $= J_0(1 + ((n + 1)/n)J_0 K_3 t)^{-(3n + 1)/(n + 1)}$
(4) Cake filtration		$(1/J)^n = (1/J_0)^n + K_4 V_p: (1/J)^{n+1} = (1/J_0)^{n+1} + ((n + 1)/n)K_4 t$

$K_1$ ,  $K_2$ ,  $K_3$ ,  $K_4$ : experimental constants; *n*: flow behavior index.

*Source:* Lozano et al. (2000). (with permission)

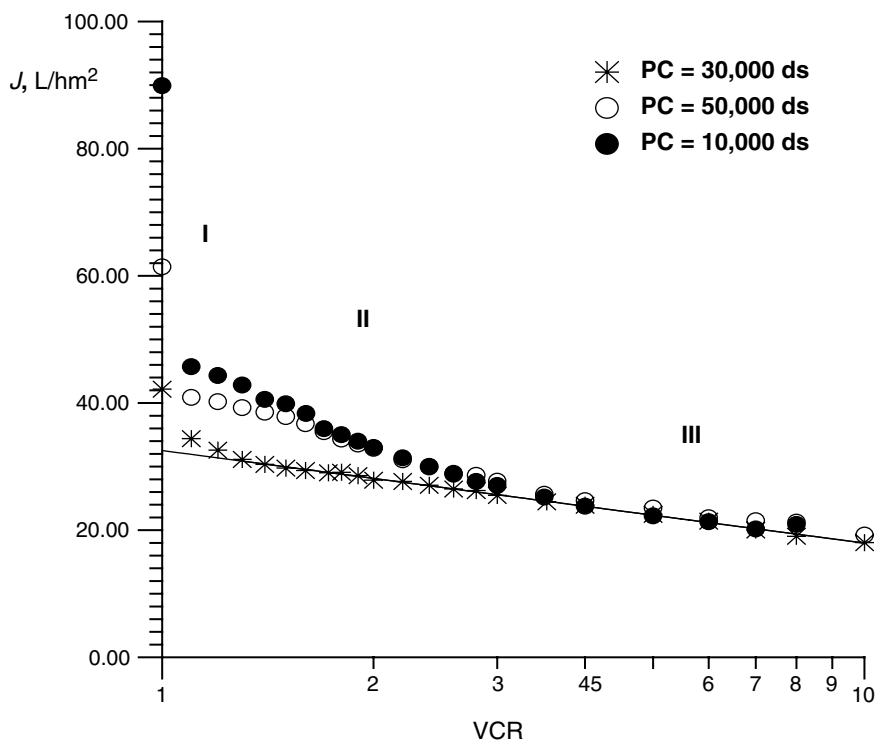
after a few minutes of operation. This effect is commonly attributed to the build up of the concentration polarization/gel layer. During the UF of apple juice in the mass transfer region, a 60% increase in  $\Delta\text{PTM}$  was reflected only as a 5% increase in  $J$ .

Acceleration of the fruit juice retentate near the membrane surface removes the accumulated macromolecules, thereby reducing the effect of concentration polarization gel layer. Due to the low diameter of the hollow fibers, high-tangential velocities can be obtained at laminar rates. Equation iv in Table 2.8, fit reasonably well for these types of membranes:

$$J = J_0 - B \ln(\text{VCR}) \quad (2.3)$$

where  $J_0$  is the initial permeate flux, and  $B$  is a constant, which depends on the system, operating conditions, and juice properties. Decrease of flux with concentration is nonlinear, and changes in the rate of permeation were better followed when plotted against  $\ln \text{VCR}$  (Fig. 2.24). The rate of flux decrease  $J$  could be divided into three periods. The first period, characterized by a rapid decrease in  $J$ , occurred in a few minutes.

During the second period (up to  $\text{VCR} = 3$  approximately) the variation of  $J$  is unstable, depending on fiber cut-off. Then  $J$  approached a “linear” steady logarithmic decrease with  $\text{VCR}$ . This behavior could be explained by considering the resistance to flux as two separate additive resistances in series: (i) the membrane resistance ( $R'_m$ ); and (ii) the concentration polarization/gel layer resistance ( $R_p$ ). During the first period  $R_p$  increases very fast reaching a value equivalent to that of  $R'_m$ . In the second region, the  $R'_m$  value is still an important



**Figure 2.24.** Decrease of permeate flux with  $\ln \text{VCR}$  for hollow fibers with different MWCO. Full line represents Eq. (2.4). Reprinted from *Lebensm. Wiss. u. Technol.* 27: 7–14, Constenla, D.T. Lozano, J.E., Predicting stationary permeate flux in the ultrafiltration of apple juice. (copyright) 1996, with permission from Elsevier.



component of the total resistance and  $J$  is not completely independent of the properties of the fiber. Finally, during the last period  $R_p$  is dominant and the cut-off of the hollow fiber becomes irrelevant.

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