FILTRATION

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Learning Outcomes. *The reader will understand the major types of filtration systems that are currently used in the winery. Knowledge of what filter to use for what purposes and the limitations of filtration are provided. The reader will gain an understanding of the role of microorganisms and macromolecules in wine production and the importance of preserving those by understanding when and how to filter.*

Particle Size Distribution

Deformable and Non-Deformable Particles

Molecular filtration

Perpendicular vs. Cross-flow filtration

Absolute vs. Nominal Filtration

Filtration Difficulties

Precoating and Body Feeding, Plate and Frame Filtration

Sterile Membrane Filtration

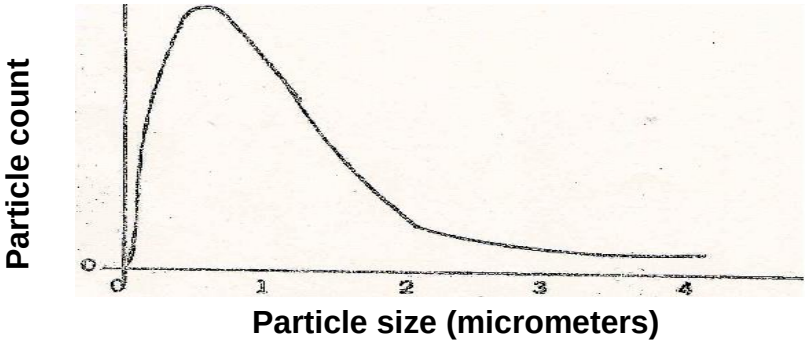
Wine Filtration and Macromolecules

Traditionally, filter porosity has been defined in terms of the physical dimensions of particulates removed, the reference being the micrometer (syn: µm or micron). Micrometer is a metric unit of measure equivalent to 39 millionths of an inch. The unaided eye can detect particulates larger than about 100 µm. Lactic acid bacteria and yeast range in size from near 0.5µm to slightly over 1 µm, respectively.

Particle Size Distribution in Juice and Wine

In any solution, the particles in suspension have a number of properties that affect the way in which they are retained by a filter. The smaller the particle, the more difficult it is to remove it from solution. One important characteristic of suspended particles is their size distribution (see Figure 1).

Figure 1. Particle Size versus Relative Frequency for a Typical Distribution of Particles in Suspension



Although the range of particle sizes in a suspension is large, a substantial number of particles are well below one micrometer (µm) in size. The average diameter of yeast (*Saccharomyces*) is about 1.2 µm. The particle size distribution in juice or wine is nonsymmetrical in nature. The preponderance of particle sizes is clustered rather closely toward the smallest particles in the distribution, with the population of larger particles being minimal.

Deformable and Non-Deformable Particles

Another important characteristic of suspended particles in solution is their mechanical nature. Particles can be classified as either *non-deformable* or *deformable*. Non-deformable particles are those which retain their shape.

In wine, the principal non-deformable particles may be diatoms. Diatoms, or diatomaceous earth, are often added uniformly before and/or during filtration to increase the filtration surface area. Because of their rigid nature and geometry,

they themselves act as a filtration medium. Using various grades of diatomaceous earth (D.E.) is a means of controlling the size and amount of particles a pad type of plate and frame filter will retain (see discussion of precoating and body feeding).

The vast majority of particles in juice or wine are the gelatinous or deformable materials. These include yeast, bacterial cells, and many colloids including fining agents. Because of their elastic nature, they are capable of spreading over a larger surface area. Hence, they are often active in blocking filtration, due to their spreading or matting effect.

Table 2. Nature of Wine Particulates

Crystalline	Amorphous Materials and Colloids	Fibrous Materials, etc.	Microbes
Potassium bitartrate	Proteins	Cellulose	Yeast
Calcium tartrate	Tannins	Case lint	Bacteria
Calcium oxylate	Pectins	Starch	Molds
Cork	Glucans	D.E.	
Calcium mucate	Metal complexes		

A third property of suspended particles is their tendency to agglomerate or flocculate (glue together). Many suspended particles will adhere if they come in contact with a similar particle. The result is a single larger particle where, formerly, there were two.

This tendency can be put to practical use in that larger particles may precipitate naturally or through the addition of fining agents. It is easier to remove larger particles by filtration than smaller particles. Thus, natural settling, prefiltration and/or fining is often a desirable means of increasing filterability.

Molecular filtration

Compared with particulate dimensions, size at the molecular level is relative to weight (or mass). Daltons (Da) or kilodaltons (kDa) identify the mass (or

molecular weight) of a molecule(s) compared to that of a hydrogen atom. As such, a molecular mass of 500 Da corresponds to solute (material to be removed) dimensions of approximately 1nm. A membrane filter porosity of 1.0 μm is equivalent to around 500,000 Da.

Macrofiltration (such as filtration pad or diatomaceous earth or both) removes microbes and larger suspended materials, depending upon the nominal or absolute porosity of the filter matrix, whereas microfiltration effects clarification in the range of 0.1-0.2 μm.

Ultrafiltration (UF) removes soluble macromolecules such as pigments, tannins, polysaccharides as well as colloidally-suspended substances in the range of 1,000 - 10^6 Da.

Porosity can be defined in terms of molecular weight cut-off (MWCO). For example, a UF membrane with a MWCO of 100,000 will remove solutes/colloidal of MW >100,000. However, a clear distinction should be made between MWCO and absolute porosity as defined in sterile membrane applications. As is the case with nominal filters, MWCO represents an approximation and should not be taken to define a specific solute mass.

Table 1.

Separation Mode	Molecular Mass (Da)	ΔP
Ultrafiltration	500-25,000	<200
Nanofiltration	<150	<250
Reverse Osmosis	<500	>200

Nanofiltration serves as a loosely-defined bridge between UF and high retention reverse osmosis (RO). Unfortunately, a universally accepted definition, based upon solute size retention or removal is lacking and varies depending upon whether the separation is viewed from a regulatory or applications mode. Section 24.248 of the Federal Register (Title 27 CFR) summarizes these distinctions in terms size of solute removed and the maximum pressure (ΔP) across the membrane to achieve separation

Nanoseparation uses membranes to preferentially pass monovalent (single charge) while excluding divalent ions. On a molecular-size (mass) basis this refers to solutes between 500 and 1,000 daltons. Thus, nanofiltration is viewed as a extension of reverse osmosis and is often referred to as "loose RO." It's primary application is to reduce levels of compounds such as 4-ethyl phenol/4-ethyl guaiacol, haloanisoles, smoke taint metabolites, etc., which lie in the molecular weight range of 100-150 daltons. Unfortunately, there is generally some collateral loss of wine flavor and character associated with the separation.

Because of the membrane's near impermeability to wine flavor, color and tannin, "tight RO," or hyperfiltration, is used for the removal of small molecules such as alcohol (MW=46), and acetic acid (MW=60) which pass readily into the permeate. These membranes can also be used to remove ethyl acetate (MW= 88) and components such as 4-ethyl phenol (MW=122). The passage of these molecules is slower and thus adds to processing costs.

Molecular mass, by itself, does not predict ease of solute separation.
For example, the polar properties associated with intramolecular charge distribution impact mass. The electonegativity associated with the oxygen creates a slight negative charge whereas the hydrogen atoms carry a slight positive charge thereby creating molecule with two poles; positive and negative. The electrical dipole created by the charge distribution interacts with other similarly charged molecules (via hydrogen bonding) in creation of a hydration

shell or “skin.” It is estimated that each water molecule reacts with the equivalent of 500-900 Da of hydration thereby increasing the effective “size” and, hence, physical properties of a single molecule of water by many-fold. Hydration also drives other components of the wine matrix to aggregate in formation of colloids. Thus, anthocyanins and other phenolics exist in colloidal complexes many hundreds of times larger than their formula weights would predict.

Perpendicular-vs. Cross-flow filtration

Depending upon the goals, filtration can vary from little more than removal of visible debris to microbes and beyond. With the advent of cross-flow filtration, winemakers can clarify relatively high solids and colloidally-laden wines. Mechanistically, filtration can be divided into forms:

- Perpendicular-flow
- Cross- or tangential-flow filtration

In perpendicular flow particulate-laden suspension approaches the filter media head-on where those solids larger than the filter matrix’s nominal (or absolute porosity) are retained. Filter media can be divided into two types: diatomaceous earth (DE or powder) and paper/pad. While the physical nature of each varies, the operational principles of clarification are the same. Based upon the physical properties of the filter matrix and particulates to be removed, 3 types of filters can be employed:

Screen or pre-filters are occasionally employed to reduce levels of relatively large particulates *en route* to pad or DE filters. Their primary application is to reduce high solids loads that would lead to premature plugging of downstream filter(s). Mechanically, screen filters trap debris on the surface of the upstream side and thus relies (primarily) on direct interception of particles larger than screen porosity. In practice, most winemakers opt to use either conventional gravity clarification or selective fining agents to achieve clarity sufficient to operate the primary filter directly rather than reliance on another tier of filtration.

Compared with perpendicular flow filtration, the feed stream in cross-flow approaches the membrane tangentially, rather than head-on, and is separated, at the filter surface, into two product streams: the permeate or, that component that passes through the membrane, and the retentate, or concentrate, enriched by those solutes and/or suspended solids which, by their physical nature, are rejected at the barrier surface. Conventional perpendicular flow technology can result in rapid plugging of membranes, a reason for the interest in cross-flow. As with any filtration, the driving force for separation is the pressure differential between the feed and permeate-side of the membrane barrier. In this case, however, directional flow of wine/juice relative to the membrane surface, creates a peripheral turbulent flow, or "eddy-effect" which dislodges particulates thereby minimizing membrane plugging/ fouling. Those solutes/solids not passing through the membrane are swept away and returned to "feed tank" via a circulation loop. Despite the self-cleaning effect, retentate solids levels eventually become sufficiently high that the system requires regeneration. Unlike perpendicular-flow membrane filtration, back-flushing, in cross-flow applications, is standard operating procedure for dislodging trapped solids. In most units produced today, back-flushing is part of the automated filtration program.

Contemporary cross-flow separation/filtration applications range by more than five orders of magnitude from particulate (micropore) to sub-micron applications; ultra-, nano- and hyper- ("tight") RO.

Figure 2. Overview of Filter Types

- **DE filters**
- **Pad filters**
- **Membrane filters**
- **Cross-flow filters**

Microfiltration, ultrafiltration, and reverse osmosis

Absolute vs. Nominal Filtration

An absolute filter is a geometrically regular, porous matrix that retains particles on its surface primarily by a sieving mechanism. The filter's pore size is controlled in the manufacturing process. Filtration through such a filter is inherently absolute, in that anything larger than the pore size is retained on the filter surface.

These are the *membrane*-type filters used in the wine industry as a final filtration, just prior to bottling, for the removal of wine microorganisms. The advantages of such a filter are summarized as follows:

- It is possible to derive a specific rating of membrane efficiency independent of flow rate and pressure differential. Therefore a winemaker can be assured that no microorganisms larger than the pore diameter will travel through the filter if the filter is properly functioning. Most winemakers attempting to remove yeast use a membrane filter with a 0.6 µm pore diameter, while lactic acid bacteria are generally retained by a 0.45 µm membrane filter.

- Owing to the homogeneous nature of the membrane, no media migration or sloughing of the filter occurs. Thus, no particles larger than the membrane's pore diameter, or pieces of the membrane material itself, will travel downstream.

- Since membranes are very thin (membrane thickness = 150 µm), there is no possibility of microbial growth within the inner layers. Coupled with this property, there is reduced product loss.

- Successive layers of larger particles may act to prevent the passage of particles smaller than pore diameter.

The following points are disadvantages of the absolute-type filter:

- Because of surface retention, membrane filters have a low "dirt-handling capacity." This is especially true of particles with diameters approximately equal to those of membrane pores. Therefore only "clean" wine should be filtered through these units for the purpose of removing microorganisms.

- Not all small particles (with diameters less than pore size) will pass readily through. Many may be retained in the port passage, hence blocking the flow.

Nominal filters are those with a relative range of pore sizes. The most common nominal filtration is a depth filter such as a filter pad. In depth filtration, the separation of solids from the liquid phase takes place inside the filtration medium only. The filtration medium consists of numerous tortuous channels of all diameters and configurations. All the channels vary in diameter from the upstream to the downstream side. The particles float at random through the channels and, at some point, impact on the walls of the channel and are retained by entrapment or adsorption.

As the particles are deposited in the depth filter, its retention capacity increases. This increases the flow resistance and the differential pressure. Eventually, this results in complete blocking. Disadvantages of depth filtration include the following:

- Media migration can occur. This refers to the tendency of filter media fragments to slough off during filtration. This problem is increased in cases where the wine to be filtered encounters the filter as a surge, rather than at a uniform flow.

- Microbial growth within the filter matrix may become a problem, especially in long filter runs. Under proper conditions, organisms may reproduce within the filter and successive generations will penetrate deeper into the matrix. The result is contamination of the filtrate (wine that has passed through the filter).

- A certain amount of product may remain within the filter matrix after filtration. In the wine industry, the filter is usually “blown out” with nitrogen, and the trapped wine is transferred back to the feed tank.

Since the depth filter can retain particles throughout its matrix, rather than solely on its surface, it will filter many times the material that the absolute-type filters

can process. Further, owing to its principle of adsorption, this filter will retain particles smaller than its flow passages.

Because of the nature of depth filtration, an absolute particle retention rating is difficult. These filters are assigned a *normal rating*. This is usually a particle size, above which a certain percentage (usually 98%) of particulates will be retained. It is important to note that this rating is valid only under strictly defined conditions of flow, temperature, pressure, and viscosity. Change in any parameter will affect particle retention. The so-called *sterilizing pads* are depth filters especially made to have a uniform porosity. These pads, however, can only remove yeast under a specific set of conditions, such as flow rate and differential pressure across the pad.The depth filters in common use in the wine industry include the pad filter-like plate and frame filters, as well as pressure leaf or cake filters.

In order to improve the filtration characteristics of this system, the wine industry uses diatomaceous earth (D.E.) for pre-coating of the screen or filter pads, as well as for a continuous proportioned body feed throughout the filtration cycle. By selecting the particle size of the D.E. used, different fineness of filtration can be achieved, from rough filtration to polish filtration.

Filtration Difficulties

A number of factors determine the filterability of wines, including the following:

- Grape variety
- Fruit rots
- Fruit type
- Season
- Processing
    - Enzymes
    - Fining, pre- and post-fermentation
    - Residual fining agents
    - Yeasts
    - Bacteria
    - Temperature
    - Carbon dioxide
    - Thermal treatment

Polysaccharides and Filtration

Polysaccharides, including pectins and glucans, are deformable particles that may be present colloidally in juice and wine where they can impede filtration. In alcoholic solution, both pectins and glucans are unstable, forming characteristic gelatinous aggregates.

Glucans are produced as a result of *Botrytis* growth on grapes, as well as from spoilage lactic acid bacteria. Presumptive identification is based upon gel formation, following the addition of ethanol. Two methods are used to decrease the level of glucan contamination (see Zoecklein et al., 2005).

Pectins

Pectins are structural components of plant cell walls that may impede clarification and, with time, develop haze or sediment in wine. If pectin is present, based upon the following test, the addition of pectolytic enzymes to a laboratory sample and subsequent precipitation test is recommended. Such remedial action is slow and costly. Proactive enzymatic treatment of must/juice is recommended. It should be noted that there should be less than 48 hours between filtration and membrane filter due to the possibility of pectin polymer reformation.

Starch

Starch can cause hazes in apple juice and cider, affecting clarity and filtration. For a procedure to identify starch haze, see Zoecklein et al. (2005).

Precoating and Body Feeding, Plate and Frame Filtration

Plate and frame filters consist of a number of plates and frames, corresponding in size and shape, which are arranged alternately, and which are supported on a pair of rails. The plates have a ribbed or waffle surface to facilitate the flow of filtrate. They may be constructed of stainless steel or plastic.

The feed channel in this filter is formed by corresponding holes in each plate and frame that register together when the filter is tightened, so that they form a continuous flow path. Each frame has an opening that leads from this channel into the inside space of the frame. There is another opening in the bottom of each plate, that connects the down-flow side of the filter cloth to an outflow channel formed in a manner similar to the feed channel, and which leads to the filtrate outlet port. When the filter is in operation, liquid flows into the filter through the inlet port, frame ports, cake, filter cloths, plate ports, and out through the filtrate outlet port.

During the initial stage of the operation, the liquid is filtered through the filter pad only. Therefore, a circulation phase is needed in order to deposit a cake on the surface of the filter cloth. This is known as *precoating*. Once this is accomplished, the flow is diverted into a tank and the filtration continues while more D.E. is added to the wines as a *body feed*. D.E. is composed of the fossil remains of microscopic marine plants called diatoms. These plants extract silica from the water and form exoskeletons (shells). The skeleton remains after the plant dies. Diatomaceous earths are processed at 1500-2000°F to burn off all organic matter. This leaves a residue which is almost pure silica.

There are various grades of D.E. depending on the fineness or particle size, which ranges from about 2.5 to 38 µm. The finer particle size produces a more-polished filtration. The amount of D.E. needed to deposit an effective precoat depends on the flow characteristics of the filter, the type of screens and filter pads used, and the pump characteristics. The most effective amount can only be determined by actual experimentation

The body feed prevents rapid plugging of the filter by providing a continuous supply of new porous filter elements. As a general rule, 10 to 15 lbs. of D.E. per 100 sq. feet of surface will be ample for a 1/16 inch precoat, if the cake is evenly distributed.

Sterile Membrane Filtration

Depth filtration is usually the first step in the filtration process. If membrane filters are used, they must be preceded by a filter system which performs 99.9% of the work. Additionally, membrane filters must be bubble tested, before, (possibly during) and after each day's run.

If the membrane fails the integrity or bubble test, everything produced between that time and the previous test is suspect.

The "bubble point" is that gas pressure at which the surface tension of water in the capillary pores of a saturated filter is overcome and gas is allowed to pass through the pores. It is directly dependent on pore diameter. The bubble point test is a final check and will determine if leaks are present anywhere in the filtering system. The bubble point test should be run immediately after the holder is assembled and while the filters are still wet. It also should be used before the daily run and directly afterward as a means of checking the integrity of the system.

Sterile filtration may also be achieved with sterile filter sheets. Throughout the run, maximum flow rates must be rigorously observed. If a filter requires sheets with internal holes, close attention should be paid to ensure that there is no bypassing of the sheet in the vicinity of the holes. The final filter, be it sheet or membrane, must be sterilized before each bottling run.

At the end of the run, winemakers should take out each sheet and look at the downstream side of the pad. Sheets can be weakened on installation or use.
A major source of ruptured filter sheets is back-pressure shock caused by no upstream support and rapid closing of valves, shutting off of pumps, pulsation of pumps, etc. In any of these cases, chances are good that an experienced eye looking at the used sheet will see the characteristic flaw or a dark line.

Every square millimeter of material in contact with the wine, from before the final filter to the bottle, must be thoroughly sterilized. To simplify this, the entire system should be analyzed. All fittings which are not absolutely necessary should be removed. Sterilization should be done at the end of a run, to prevent organism buildup. The procedure should be repeated before the next run.

Either hot water (steam) or chemical sterilization should be used before the run and equipment must be thoroughly cooled and/or rinsed with sterile water.

Wine Filtration and Macromolecules

Debate continues as to whether wine filtration, in any form, changes the sensory character of a wine character. Those choosing not to filter wines prior to bottling should review carefully the following:

- Microbiological content
- Substrate availability
- Possibility for bottle inconsistency, control of aroma / flavor / mouthfeel
- Possible decreased stability of total and free sulfur dioxide post-bottling

The wide range of soluble flavor and aroma-active compounds present in wine relative to the diameter of a 0.45 µm pore might suggest that the effects of filtration may be negligible. Sensory impacts have been difficult to quantify due to the number of variables including wine composition, age of the wine at filtration and evaluation, and the subjective nature of sensory assessments. Soluble species are, likely not directly removed by conventional macro- and micropore filtration, colloidally-suspended macromolecules which impact mouth feel maybe. These may be present as large aggregations of polysaccharide, manoprotein or protein-phenolic complexes (500+ kDa) where they may play a role in the wine's textural/structural presentation. Interactions between macromolecular species and lower molecular weight volatile compounds may, in part, account for apparent aromatic changes noted after sterile filtration.

During aging, phenols bind together or polymerize. As they do, their molecular weight and size increases. Filtration can remove some of these. Such removal depends on a host of factors, including the age of the wine. Older red wines have a much greater percentage of polymerized phenols and, thus, can show a much greater negative effect from filtration. That effect is in the form of some color and body or volume reduction (Tables 2 and 3).

Table 2. Effects of Different Types of Filtration on the Chemical Composition of a White Wine (results in mg/L)

Parameter	Control	Coarse DE	Fine DE	Clarifying filter sheet	Sterilizing filter sheet	Membrane 0.65 µm
OD 420	0.084	0.087	0.083	0.079	0.080	0.078
Tannins	71	69	68	67	68	66
Total polysaccharides	570	540	517	521	518	454
Higher alcohols (total)	317	312	312	308	309	291
Higher alcohol acetates (total)	3.5	3.5	3.5	3.4	3.2	2.9
Volatile fatty acids (total)	14.3	14.0	12.8	13.8	13.7	12.3
Ethyl esters of fatty acids (total)	4.3	4.2	4.0	4.4	4.0	3.8

Table 3. Effects of Different Types of Filtration on the Chemical Composition of a Red Wine (Serrano and Paetzold, 1994)

Parameter	Control	Coarse DE	Fine DE	Clarifying filter sheet	Sterilizing filter sheet	Membrane 0.65 µm
Free polysaccharides (mg/L)	426	420	389	380	385	342
Total polysaccharides (mg/L)	650	630	607	625	620	562
Phenol	41	40	39	40	39	37

compound index (D280)						
Tannins (g/L)	2.7	2.6	2.4	2.5	2.4	2.3
Total anthocyanins (mg/L)	252	243	225	240	230	208
Color Intensity	0.53	0.54	0.62	0.59	0.59	0.57
Hue	0.81	0.79	0.81	0.78	0.80	0.80

Filtration reduces the particulate load and, in the case of sterile applications, viable microorganisms. Hence, those opting not to filter must be aware of the potential for post-bottling biological instability. Addition compounds and technologies such as Velcorin™ and supercritical CO_2 injection, high pressure and High Temperature Short Time (HTST) pasteurization are being perfected as alternatives to sterile bottling without filtration.

Practical Summary of Winemaking Filtration Issues

- Prefiltration fining may be advisable to improve wine filterability.
- Conducting a filterability test can be an asset in determining what filtration medium to use.
- Membrane filters with a pore size of 0.8 µm are used to remove yeast, while those with a pore size of 0.45 µm will remove lactic acid bacteria prior to bottling.
- Absolute (or membrane) filters act predictably in their filtration, but plug easily, so they are best used when the wine is already quite “clean.”
- Depth filters catch particulate matter through entrapment or adsorption. They are less predictable in their actions than are absolute filters, requiring very specific conditions, but plug less easily.
- The use of diatomaceous earth (D.E.) in precoating a filter, and mixed in with the wine during filtration, may improve filtering success.

Study Questions

1. What are the three main types of filters?

2. How do the three main types of filters differ in their ability to remove deformable particles? Non-deformable particles?

3. How does one decide on which filtration system to use?

4. Why is a pectin test a good idea before filtration is undertaken?

5. What are the major advantages of filtering a wine? What are the disadvantages?

6. Discuss the possible relationships between wine filtration and the loss of macromolecules.

7. Why has it been so difficult to determine the relationship between filtration of red wines and sensory advantages or disadvantages?

8. Define body feeding and discuss its practical importance.

9. Why may the time between depth and membrane filtration be important?

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