MBAA TQ vol. 42, no. 4 • 2005 • pp. 332–338

Beer Stabilization Technology—Clearly a Matter of Choice

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ABSTRACT

The mechanism of formation of nonbiological haze in beer is considered with reference to the key raw material and process stages that impact polyphenol and protein haze precursors. Strategies to minimize their formation during beer production are also suggested. The options for achieving good colloidal stability are considered, including a novel upstream product employed in the brewhouse toward the end of wort boiling. This has the advantage of improving wort clarity and cold wort runoff volume, as well as increasing the shelf life of the beer. More conventional approaches employ additions after fermentation, prior to and post-diatomaceous earth-filtration. The mechanism of action and typical dosage rates employed are illustrated along with the major benefits of each class of stabilizing agent. A combined product is also considered in the context of simplifying colloidal stabilization and introducing the concept of a balanced reduction of both the major classes of haze precursors—polyphenols and proteins. This approach provides the most effective colloidal stabilization. Seven key control steps to achieve good product shelf life are highlighted.

Keywords: beer, colloidal stability, haze, polyphenol, polyvinylpolypyrrolidone (PVPP), protein

SÍNTESIS

Se discute sobre los mecanismos de la formación de turbiezas no biológicas con respecto a las principales materias primas y las diferentes etapas de producción que afectan los precursores de turbiezas polifenólicas y proteínicas. Se discuten las opciones para alcanzar una buena estabilidad coloidal, incluyendo un producto novedoso utilizado al final del hervor del mosto. Además de mejorar la estabilidad de la cerveza, el uso de este producto también mejora la claridad del mosto y aumenta el volumen del mosto frío. Productos convencionales son usados después de la fermentación, antes de o después de la filtración por kieselgur. Se informa sobre el mecanismo de adición del producto y las proporciones típicamente utilizadas, además de ilustrar los beneficios principales de cada clase de agente estabilizador. También se considera un producto combinado para simplificar la estabilización coloidal; se introduce el concepto de una reducción balanceada de ambas clases de precursores de turbieza—polifenoles y proteínas—para así optimizar la estabilización, Se dan siete pasos claves para alcanzar una buena estabilización.

Palabras claves: cerveza, estabilidad coloidal, turbieza, polifenol, polyvinylpolypyrrolidone (PVPP), proteína

Introduction

The Need for Colloidal Stabilization of Beer

It is said "people drink beer with their eyes". An important index of beer stability is the visual appearance of the product. With the exception of a few well-known examples, such as Weiss beer, consumers associate a star-bright product as a mark of freshness. While a beer is likely to deteriorate in terms of flavor before the appearance of haze, most consumers will probably notice the latter first (9)! The challenge is to present this visually appealing product to the beer drinker, sometimes months after manufacture. This (longer) time frame is driven in many mature beer markets by a shift from draft beer consumption in bars to at-home consumption from bottles and cans. Add to this the growth of premium beers from both microbreweries and imports, and the time between manufacture and consumption increases (3). In this context, a shelf life of 6 months to 1 year is now common.

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Based on a paper presented at the World Brewing Congress 2004, San Diego, CA, July 2004.

DOI: 10.1094/TQ-42-0332

Mechanism of Nonbiological Haze Formation

When talking about haze, it is important to distinguish colloidal haze from microbiological haze; the latter being rare due to the significant improvements in the design and operation of breweries during the last century. Colloidal or nonbiological haze comprises mostly proteins, polyphenols, carbohydrates, and some minor constituents (1,2,7). The mechanism of haze formation is an interesting topic and still the subject of academic investigation. Karl Siebert's group at Cornell University (16–18) has undertaken significant research in this area and has proposed that the two major constituents—haze-forming proteins and polyphenols—have specific structures that give them a propensity to interact and form colloidal particles (17,18). The haze proteins have regions rich in the amino acid proline, to which the polyphenols attach, as shown in Figure 1. Interestingly, proline is the only amino acid in wort not consumed by yeast during fermentation since it does not have a permease to allow transport into the cell. The structures and

nomenclature of the haze-active proteins and polyphenols have been reviewed in depth by both Bamforth (2) and McMurrough and colleagues (7), to whom the reader is directed. Another model of chill and permanent haze development by O'Rourke and colleagues (12) proposed that the driver in haze formation was the oxidation of flavanol oligomers (sometimes also called flavanoids) during beer storage. A kinetic model of this oxidative polymerization was developed by Rehmanji and colleagues (13), which related the rate of chill haze development to the conversion of flavanols into tannoids. As monomeric and dimeric flavanols such as catechin and prodelphinidin polymerize, they have the capability to bridge a number of proteins, forming a larger colloidal particle. This is first only visible as a chill haze because of the relatively weak hydrogen bonds linking the two, as shown in Figure 2. With further polymerization, larger polyphenol molecules (termed tannoids) are formed, which are more tightly bound to the haze proteins, giving rise to permanent haze. The tannoids are considered to be intermediates in the oxidation of the flavanols to true tannins (7). O'Rourke and colleagues (12) also showed that the simple flavanols, such as catechin per se, were not capable of haze formation, but that same molecule after oxidation did produce haze, implicating the important role of oxygen in colloidal instability (Fig. 3). Since molecular oxygen is usually not detectable in packaged beer, the same role can be attributed to oxidizing agents in the beer (2).

More recently, Mitchell and colleagues (8) identified 20 compounds in beer, including glycollated and nonglycollated proanthocyanidin (flavanol) monomers, dimers, and trimers. No molecules with a higher degree of polymerization were detected by the liquid chromatography–mass spectrometry (LC-MS) procedure. Many authors consider the oxidation of the dimers to be the key factor in the development of chill haze in beer (2,6,7,13).

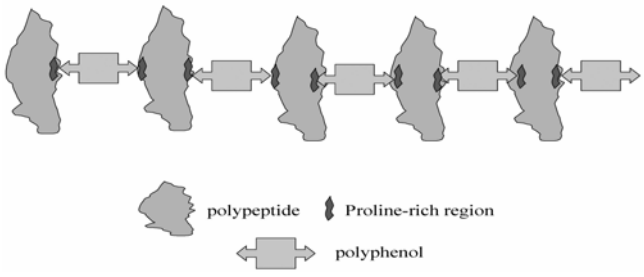

Figure 1. Model for protein–polyphenol interaction. Siebert and Lynn have proposed that the two major constituents—haze-forming polyphenols and proteins—have specific structures that give them propensity to attach and form colloidal particles (16). Polyphenols are depicted as having two ends that can bind to protein. Proteins are depicted as having a fixed number of polyphenol-binding sites.

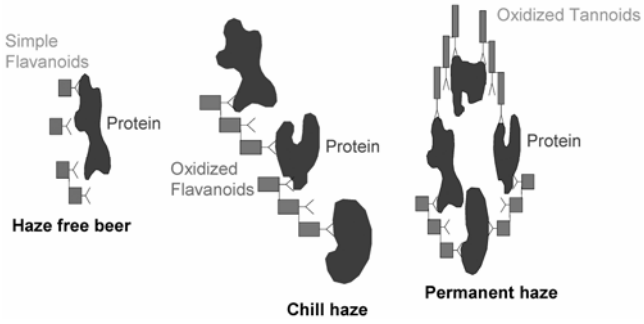

Figure 2. The model of chill and permanent haze development in beer proposed by O'Rourke and colleagues (12). Simple flavanoids (flavanols) alone are incapable of causing haze; but following oxidation and polymerization, the protein–polyphenol complexes linked by hydrogen bonds produce a chill haze (at 0°C). The hydrogen bonds are disrupted by warming to room temperature (20°C) and the haze redissolves. Tannoids are capable of forming additional hydrophobic and ionic interactions with acidic proteins, which are not disrupted by warming to 20°C and result in a permanent haze in the beer.

Discussion

Achieving Colloidal Stability

Assuming that the freshly packaged beer has no detectable tannoids present, during the initial storage of the beer, there is only a slight increase in haze (6). As the lower-molecular-weight polyphenols polymerize and increase in size, there is a significant increase in the rate of haze formation, as shown in Figure 4. Hence, effective colloidal stabilization requires the removal of the tannoids and a reduction in the level of particularly dimeric and trimeric flavanols (2). The extent of this reduction depends on the shelf life requirements of the beer, the raw materials used, and the brewing procedures employed. Use of stabilizers to achieve a target shelf life should be opti-

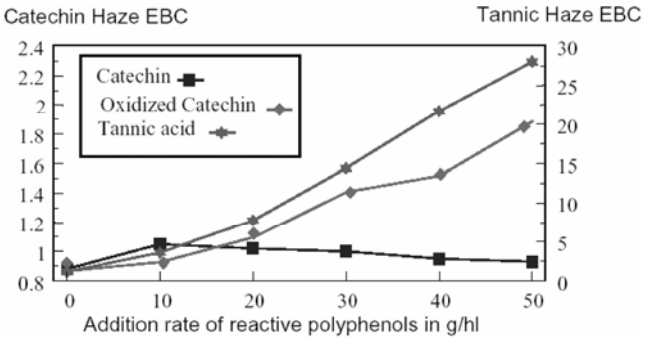

Figure 3. Simple flavanoids, such as catechin, are not capable of haze formation, but that same molecule after oxidation is an active haze producer after interacting with protein.

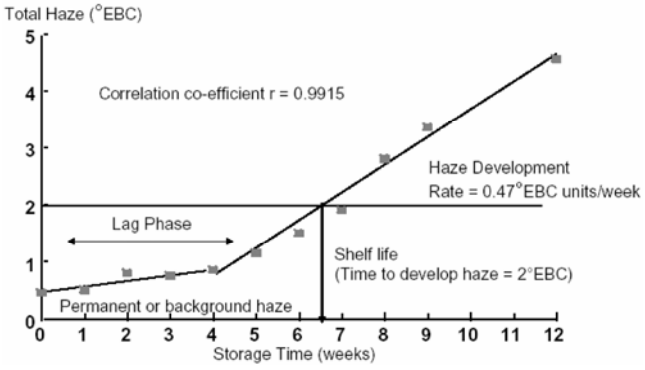

Figure 4. As the lower-molecular-weight polyphenols increase in size (polymerize), there is a significant increase in the rate of haze formation. This biphasic model of haze formation has been reported by many researchers. A well-stabilized beer is characterized by a long lag phase and a slow rate of haze development to the required shelf life of the beer.

Table 1. Raw material and process strategies to optimize colloidal stability

Process	Polyphenol reduction	Protein reduction	Process optimization
Raw material selection	Low proanthocyanidin malt Hop extract	Low protein (N) barley	Low malt modification Coarse grind of malt
Brewhouse	High adjunct ratio Avoid weak worts	Mashing process, pH, and temperature Good hot break Cold wort filtration	Vigorous kettle boil for >60 min Avoid excess mineral salts
Fermentation/maturation	Rapid onset to fermentation	Early yeast removal	Minimum 7 days of maturation at –1°C
Filtration			Low solids count Filter at –1°C Avoid O_2 pickup

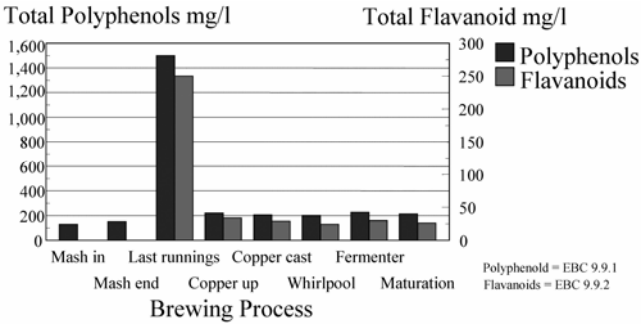

Figure 5. Change in polyphenols and flavanoids during brewing. In the brewhouse, avoiding the use of weak worts (<1.5°P) can play an important role in stabilization, since these contain very high levels of polyphenols.

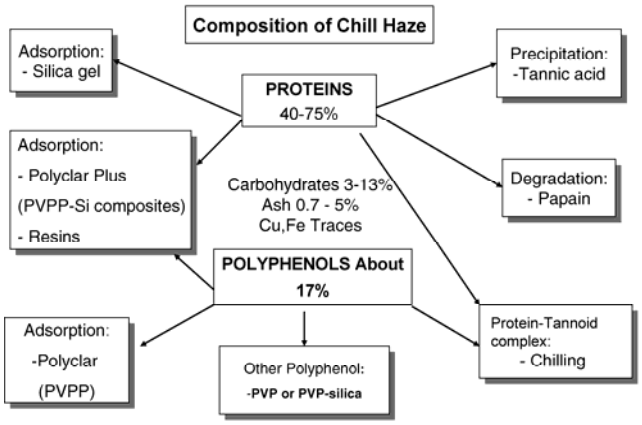

Figure 6. Options for colloidal stabilization of beer. The main stabilization options are highlighted in terms of their mechanistic actions. Some of the advantages and drawbacks of each are summarized in Table 2. Si = silica.

mized and not be excessive, which is expensive and may detract from beer quality. This equally applies to the reduction of the haze-active protein precursors.

Considerable academic and industrial research has gone into investigating both brewing raw materials and procedures and their impact on beer colloidal stability; only a brief summary can be attempted here (Table 1). For fuller reviews, readers are directed to articles by Anger (1), Bamforth (2), McMurrough and colleagues (5–7), and O'Rourke (9). At each stage, the most important thing is to pick out a few key strategies that can be adopted to reduce the formation and passage of haze precursors to the next step of the brewing process. In the brewhouse, avoiding the use of weak worts can play an important role since these contain very high levels of polyphenols that can pass into the fermentation vessel (Fig. 5). A vigorous kettle boil and good hot and cold breaks will promote early polyphenol–protein precipitation (10). A rapid onset of fermentation and early removal of yeast, preventing cell lysis, are important since yeast mannans (carbohydrate) can contribute to the haze matrix (1). As mentioned before, oxygen exposure after initial wort oxidation must be strictly prevented (1,11) and the beer at the end of fermentation chilled to –1°C or slightly lower during maturation. A maturation time of 7 days or more improves colloidal stability by allowing the formation and precipitation of protein–polyphenol flocs in the cold storage vessel. Having formed these low-temperature flocs, a temperature increase above 0°C would cause them to separate, so the beer must be held around –1°C into filtration and bright beer tank. With all these precautions in place, a respectable shelf life of a few months is possible for many adjunct beers. For high-malt beers in particular, and adjunct beers in which a shelf life of more than 3 months is needed, some form of stabilization treatment is usually required (C. Gopal, *unpublished data*), and it is this we turn to next.

Options for Beer Stabilization

Since the two major components of colloidal haze are the protein and polyphenol fractions, their reduction in beer prior to packaging is the obvious target. Figure 6 illustrates the most common options available to the brewer. The list is not exhaustive since it excludes some older technologies, such as formaldehyde, which were no doubt very effective but may leave behind toxic residue in beer. For a full review of the stabilization options, readers are directed to other reviews (1,2,7,9,16). Probably the most widely employed products today are silica gel for protein stabilization and polyvinylpolypyrrolidone (PVPP) for polyphenol stabilization. The other major product categories are tannic acid and enzyme preparations (papain), and these are also considered in turn. Before coming to these, we consider a "reinvented" technology—upstream beer stabilization. Upstream beer stabilization is easier to employ; the (stabilization) product is added directly to the boiling wort without the need for specialized equipment, such as slurry tanks and dosing units.

A Novel Wort Clarifier and Beer Stabilizer

A widely known but often overlooked observation is that beers with lesser clarity in package have a limited shelf life,

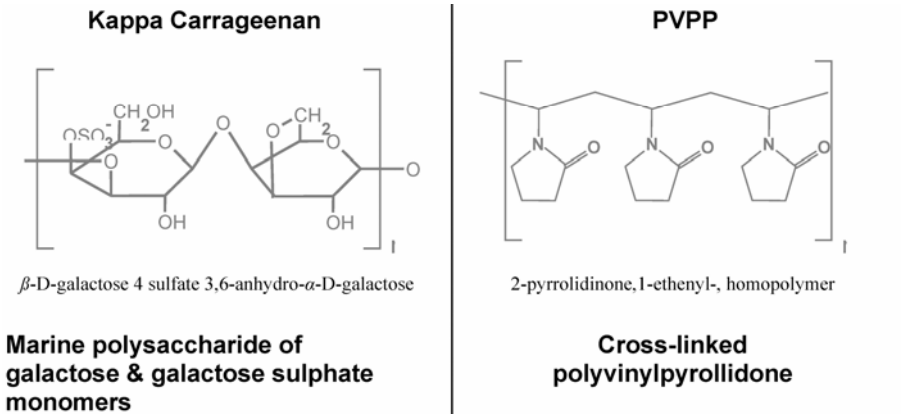

Figure 7. Combining polyvinylpolypyrrolidone (PVPP) with κ-carrageenan, Polyclar Brewbrite (International Specialty Products, Wayne, NJ) provides wort clarification and beer stabilization with a single addition to the kettle about 10 min before the end of boiling.

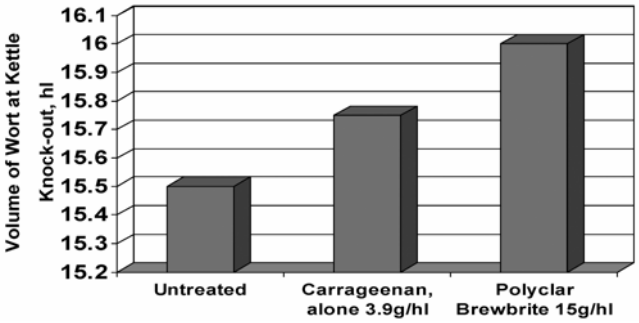

Figure 8. Increase in wort yield—commercial trial. Polyclar Brewbrite (International Specialty Products, Wayne, NJ) gave a 3.2% increase in wort yield as compared with that of untreated wort. A better solid–liquid phase separation in the whirlpool facilitates cold wort runoff, which can significantly improve process yield, without the necessity of trub recycling and its negative quality impact.

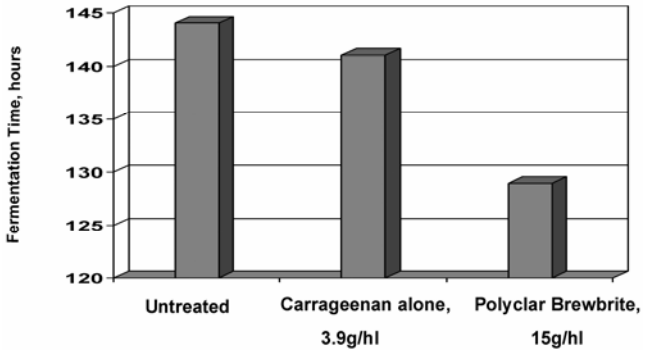

Figure 9. Decrease in fermentation time—commercial trial. Polyclar Brewbrite (International Specialty Products, Wayne, NJ) gave a 10% reduction in fermentation time as compared with that of the untreated control. The combination of carrageenan and polyvinylpolypyrrolidone (PVPP) may decrease the time to rack gravity, without impacting the overall attenuation or flavor profile of the beer.

with a tendency to rapidly become hazy. The likely mechanism is that the colloidal particles present in the fresh beer act as templates on which protein–polyphenol complexes readily attach, accelerating chill haze development. The use of isinglass finings, green beer centrifugation, and 'tighter' beer filtration are all options to improve beer clarity. Another is to clarify the wort using well-characterized fining agents such as κ-carrageenan, more commonly known as copper finings, since they are typically added 10 min or so before the end of the kettle boil. A novel stabilization agent harnesses the synergies of wort clarification and polyphenol reduction by combining PVPP with κ-carrageen (Fig. 7).

The product is added to the wort either directly into the kettle or on transfer to the whirlpool. In use, better solid–liquid phase separation in the whirlpool has been noted in trials, with increased cold wort collection (Fig. 8). A more ideal cone is often observed in the whirlpool, after wort runoff. In some instances, faster fermentation rates have also been observed (Fig. 9). The key objective is to increase the shelf life of the beer and, using this simple procedure, a shelf life of 6 months or more (Fig. 10) can be achieved without the need for capital investment in the brewery (15).

Of the stabilization options summarized in Figure 6, the most widely employed agents in current use are PVPP for polyphenol haze precursor reduction and silica gel for protein haze precursor reduction. Both have the advantages of being insoluble in water and beer and are classified as process aids; as such, their use is permitted on a global basis and specifically allowed under the German Reinheitsgebot.

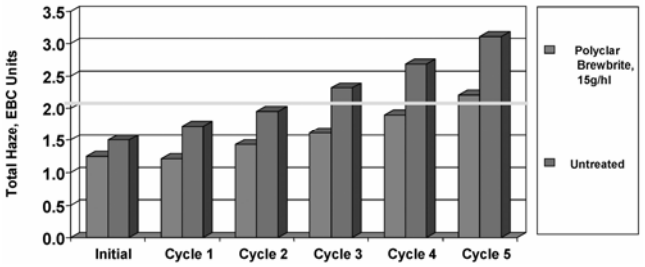

Figure 10. Analysis of packaged beer—commercial trial. Accelerated forcing test. Shelf life of beer and Polyclar Brewbrite (International Specialty Products, Wayne, NJ), in which a cycle is 24 h at 60°C followed by 24 h at 0°C. The number of cycles (to reach 2.0 EBC units) is the months of predicted shelf life. Application to wort improved initial beer clarity compared with that of the untreated control and the predicted shelf life of the beer to around 6 months. In this instance, this could be further enhanced by tighter beer filtration to produce an initial haze in the beer of approximately 0.5 EBC.

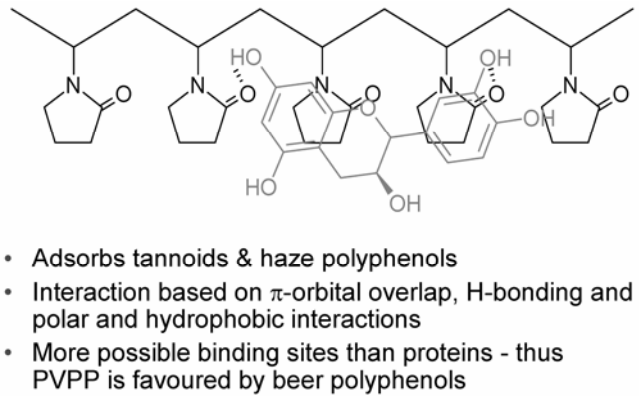

Figure 11. Polyphenol removal by polyvinylpolypyrrolidone (PVPP). The adsorption of polyphenols by PVPP is through H-bonding between the proton donor from the polyphenol and the carbonyl group from PVPP, together with π-bond overlap (delocalized electrons) and polar and hydrophobic reactions (12).

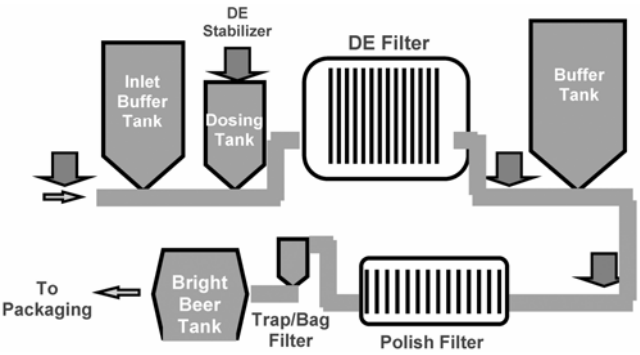

Figure 12. Schematic illustrating the possible addition points of the blended stabilizer. The highest efficacy is achieved by addition from a separate dosing tank to beer with low solids content. DE = diatomaceous earth.

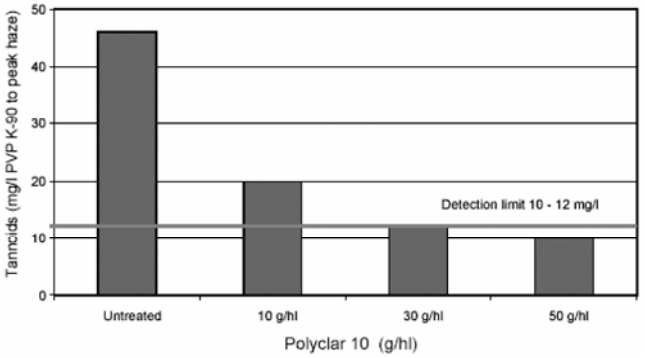

Figure 13. Tannoid reduction in an American lager treated with polyvinylpolypyrrolidone (PVPP). An addition of 10 g/hL resulted in a >50% reduction in the tannoid content of the beer. At 30 g/hL, the tannoid level was below the limit of detection of the assay.

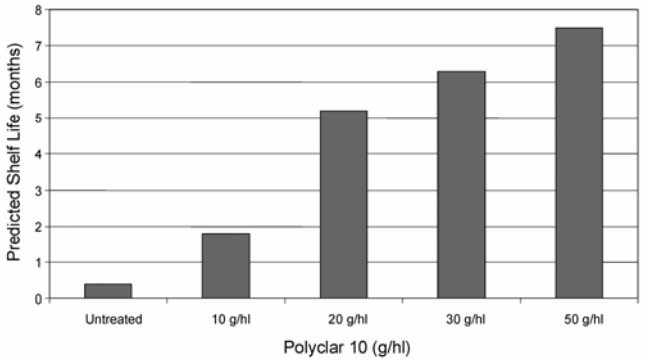

Figure 14. Increase in the predicted shelf life of polyvinylpolypyrrolidone (PVPP)-treated beer. An addition of 20 g/hL achieved a shelf life in excess of 5 months in an American lager. A longer shelf life can be achieved by the application of a protein stabilizer, such as silica gel.

They are sometimes referred to as "clean label" products, since their insoluble nature and removal from beer prior to packaging mean they are not required to be listed as ingredients.

PVPP

The unit molecular structure of PVPP closely resembles that of the amino acid proline; in fact, it can be considered as a chemical analog of polyproline—cross-linked polyproline (17). This structure facilitates its ability to quickly adsorb those polyphenols responsible for haze development—both the tannoids and polymerized flavanols discussed earlier. The adsorption of polyphenols by PVPP is through H-bonding between the proton donor from the polyphenol and the carbonyl group from PVPP, together with π-bond overlap (delocalized electrons) polar and hydrophobic reactions (13). If that sounds complicated, in fact, it can be illustrated quite elegantly in Figure 11. Polyphenol adsorption is preferentially directed to the larger oligomers first since they have the highest number of potential bonding sites to PVPP. The advantage to the brewer is that the dosage rate of PVPP can be adjusted to reduce the polyphenols most likely to cause haze. Moreover, PVPP does not have any adverse effects on beer quality, including flavor (12) and foam stability (1).

PVPP can be used either as a single-use stabilizer, or on a regenerable basis—readers are directed to reviews by Gopal and Rehmanji (3) and R. Schlenker (*unpublished data*). PVPP regeneration may become economical when large volumes of beer are being stabilized (3). The single-use grade is micronized during manufacture to give a particle with a large surface area over which the polyphenols can be adsorbed (13). This allows low, cost-effective addition rates to be employed—typically 10–20 g/hL (2.6–5.2 lb/100 US bbl). PVPP (and silica gel) is usually added at one of a number of stages in the brewing process after the end of fermentation, as shown in Figure 12. An addition of as little as 10 g/hL resulted in a >50% reduction in the tannoid content of an American lager (Fig. 13). Further increases reduced this to below the detection limit for the assay (10–12 mg of PVP K-90 [International Specialty Products, Wayne, NJ] per liter). The resulting increase in shelf life was also dramatic, with a 20-g/hL addition rate providing a predicted shelf life in excess of 5 months in this beer (15), as shown in Figure 14. Further dosage increases yield diminishing returns.

This leads us to the concept of balanced stabilization, i.e., achieving the most (cost-) effective reduction of both the polyphenol and protein haze precursors. The benefits of this "holistic" approach have been widely proposed (2) and demonstrated by many researchers (1,4,7,14,19).

Table 2. Summary of the most widely used stabilizing agents for preventing chill and permanent haze development in beer

Stabilizer	Mechanism of action	Advantages	Disadvantages	Dosage range
Polyvinylpolypyrrolidone (PVPP)	Strong bonding to haze-active polyphenols via multiple bonding mechanisms	Selective for haze-active polyphenols Single use or regenerable	Possible flavor loss if used in excess Capital cost for regeneration plant	5–40 g/hL (1.5–10.5 lb/bbl)
Silica hydrogel	Adsorbs haze-active proteins via hydrogen bonding	Selective for haze-active proteins	High usage rate	30–100 g/hL (8–25 lb/bbl)
Silica xerogel	As hydrogel	Selective for haze-active proteins Lower usage rate than hydrogel	Difficult to disperse Reduced head retention if used in excess	20–50 g/hL (5–13 lb/bbl)
Gallotannin (tannic acid)	Adsorbs haze-active proteins by hydrogen bonding	Selective for haze-active proteins Low usage rate	Beer losses if used in tank Flavor harshness if used in excess	4–10 g/hL (1–2.5 lb/bbl)
Papain	Proteolytic enzyme—degrades proteins by hydrolysis	Low usage rate Persists in product	Negative impact on head retention	1–6 g/hL (0.25–1.5 lb/bbl)

Silica Gel

Just as PVPP is the stabilizer of choice for polyphenol reduction, the same is true of silica gels for haze protein reduction. The two types—hydrogels and xerogels—differ in their water content, efficacy, permeability, and handling characteristics. Selection really comes down to finding the most appropriate one for the individual brewery situation. Their action is based on the diffusion of the proteins from the beer to the silica surface, followed by adsorption to the hydrated silica gel surface and penetration of these surface-adsorbed proteins into the silica (pores). It is the last stage that is the rate-determining step, and the pore structure plays a major role in the selectivity of the silica for adsorbing haze proteins in preference to others, e.g., the hydrophobic foam polypeptides (2,16).

Other Protein Stabilizers—Tannic Acid and Papain

Two other protein stabilizers are also widely used—tannic acid and papain. The former, usually derived from Chinese gallnuts, is a very effective means of complexing protein haze precursors—in effect, precipitating tannin–protein flocs—which can be removed from beer. Its main disadvantage is that, added after fermentation, these flocs can form a voluminous precipitate at the bottom of the maturation vessel, which contributes to beer loss and also make beer clarification more difficult. However, if tannic acid is added in the brewhouse, these advantages can be overcome.

The use of enzymes—various peptidases—extracted from the papaya fruit is also quite widely used, providing a simple and inexpensive method of reducing protein content in beer. However, unlike silica gel and tannic acid, it cannot discriminate between haze- and foam-active proteins, so beer treated with papain suffers from a progressive loss of head retention (foam stability.) In addition, papain is not inactivated by pasteurization and remains active in the beer. All the stabilization options discussed are summarized in Table 2, and the reader's attention is also drawn to the excellent reviews in the literature (1,2,9).

Composite Stabilizers

Products that combine both polyphenol and protein stabilization efficacy are also widely available and include mixtures of PVPP and silica gel, such as Polyclar Plus 730 (International Specialty Products). (3,4,14,20). These have the advantage of convenience and effectiveness—only one slurry is made up and used and efficacy is enhanced: the beer treated with the combined stabilizer provided the same shelf life at a lower overall powder addition to the beer stream (Fig. 15). This can result in longer filter run lengths (4). A full-scale filtration and stabilization trial confirmed that the PVPP–silica product, Polyclar Plus 730, gave lower filter differential pressure and a longer run length than did the constituent components (Table 3). An explanation for this lies in the capacity of the PVPP particles

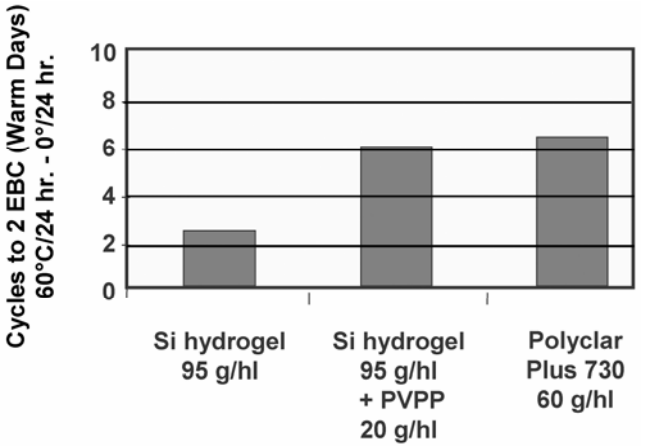

Figure 15. Predicted shelf life results—plant trial with Polyclar Plus 730 (composite 70% silica [si] plus 30% polyvinylpolypyrrolidone [PVPP]). In brewery trials, the admixture of PVPP and silica—Polyclar Plus 730—gave cost-effective efficacy of polyphenol and protein by a single addition to the beer stream without the need for specialized equipment. Balanced stabilization is efficient and cost effective.

Table 3. Increase in filter run length with combined application of polyvinylpolypyrrolidone (PVPP) and silica gel as an admixture (Polyclar Plus 730)

Stabilizer	Addition rate (g/hL)	Δp/h	Filter run time (h)
Xerogel only	36	0.49	6.4
PVPP only	13	0.35	7.4
Polyclar Plus 730	33	0.25	12.0

to help disperse silica particles (4,14) and form a more uniform and permeable bed on the diatomaceous earth (DE) filter.

After Packaging

The brewer's responsibility for beer clarity does not end after packaging the beer, since the transportation and subsequent storage of beer can have a very significant impact on visual clarity. Excessive shaking (during transportation) may result in the appearance of haze or even "particulates" before the beer reaches the customer (2). High temperatures will, of course, accelerate haze development. To quote a typical instance—one of the standard heat forcing tests in European and North American markets, incubation at 37°C, is often lower than are ambient temperatures in many developing beer markets or even the high temperatures experienced in some U.S. states during the summer months!

Conclusions

This paper covers many aspects of beer stabilization and their importance in enhancing the shelf life of beer. Beer stabilization is a vast subject, and the areas covered provide only a starting point for further investigation by the brewer. The authors recommend close examination of a number of key process steps that will certainly improve the appearance of their beers (C. Gopal, *unpublished data*) and assist them to optimize their stabilization procedures. Our recommended seven key steps to colloidal stability are as follows.

1. Avoid use of very weak wort (<1.5°P).
2. Minimize oxygen pickup throughout the brewing process (<0.1 ppm of dissolved O_2 in beer ex-fermenter and into package).
3. Cold store, transfer, and filter beer at 0°C or below.
4. Wort and beer clarity are important—optimize finings, centrifugation, and filter aid use.
5. Ensure that the tannoids are removed from fresh beer and polymerized flavanols reduced—use PVPP.
6. Balanced stabilization is efficient and cost effective—also use silica gel or a combined stabilizer, such as Polyclar Plus 730.
7. Consider beer transport and storage conditions.

ACKNOWLEDGMENT

The authors would like to thank the management of International Specialty Products for permission to publish this paper.

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