



EVALUATION OF A SUBSTITUTE FILTER MEDIUM FOR REMOVAL OF HAZE IN BEER

by

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Errata

Equation 2.2, page 18, the symbols B and P should appear as subscripts.

Nomenclature, page 107, insert:

K	permeability of the filter bed
u	filtrate flux
w	mass of solids deposited per unit area of the bed
ρ_B	bed density
ρ_P	particle density

Explanatory Note

Figures 4.6 to 4.8 represent a bed mass of 18.83 g and a pressure gradient of 180 kPa.

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SUMMARY

Diatomaceous earth (DE) is widely used in the filtration of beer to remove suspended yeast and other particulate material that can cause cloudiness or haze in the final product. The DE used has a particle size diameter of between 60 and 100 μm . However, health and safety concerns arise from its human carcinogenic classification in 1997 from Category 2 (Probable Human Carcinogen) to Category 1 (Human Carcinogen) by the IARC¹. In a confidential study² conducted at Adelaide University³, zeolite-A, a hydrated aluminosilicate of alkali earth metals, showed promising filtration capabilities when used in the removal of haze in white wine. Zeolite-A is non-toxic via oral, dermal, ocular and respiratory exposure as well as safe for the environment at disposal. An experimental study to investigate zeolite-A as a possible substitute for DE in the brewing industry was therefore undertaken. The particular zeolite-A used was selected as it was judged as nearly the same as that manufactured within the Department of Chemical Engineering, Adelaide University from naturally occurring deposits of kaolin.

Two size-grades of zeolite-A (large diameter particles of 125-250 μm and small diameter particles of 63-125 μm) were selected to cover the particle size range of widely used DE (as Celite 503⁴). These two zeolites, together with filter sand (200 μm diameter particle size) and silica were experimentally evaluated against DE in the clarification of beer simulants and commercial beer product. Flux-time experiments on each of three packed beds of each of the five filter media (3.63, 11.23, 18.83 g) with three values of pressure driving force (70, 125, 180 kPa) were carried out in a specially constructed pilot plant, initially in the laboratory and later *in situ* in a commercial brewing plant⁵. This special pilot plant, together with protocols for the preparation of media, simulated conditions and practices in current use in the brewing industry.

¹ International Agency for Research on Cancer (IARC) 1997/1998. International Diatomite Producers Association *Reclassification of Crystalline Silica*. Long Beach, CA 90803.

² Davey K R, Kadir J and Pecanek J 1997. An assessment of six (6) alternate filter media for the polishing of wine. Department of Chemical Engineering, University of Adelaide, Food Technology Research Group, *Confidential Report*. 60 pp.

³ formerly The University of Adelaide.

⁴ Marketed by FilChem Pty Ltd, Victoria.

⁵ Coopers Brewery Ltd, Leabrook, SA 5068.

The flux obtained from the small grade zeolite-A (particle size 63-125 μm) was significantly lower compared to DE, i.e. respectively, 22 $\text{mLm}^{-2}\text{s}^{-1}$ and 390 $\text{mLm}^{-2}\text{s}^{-1}$ (using 18.83 g media at 180 kPa). Large grade zeolite-A (particle size 125-250 μm) showed comparable flux properties to DE with flux rate of 290 $\text{mLm}^{-2}\text{s}^{-1}$.

Microbiological analyses were carried out initially on eight selected filter media - which also additionally included pumice, perlite and cellulose (as cotton wool) - to assess effectiveness in removing haze forming constituents from a simulated beer (yeast solution) and two home-brewed beer types. The pumice, perlite and cellulose were rejected as alternative filter media because of poor performance in haze removal. Microbiological, chemical and sensory analyses were carried out on each of the five remaining media. Results of the microbial analyses highlighted that DE and zeolite-A were the best filter media because practically all yeast cells were retained on the filter cake from both the simulated beer and the home-brewed beers.

With filtration of commercial beers using small grade zeolite-A as the filter medium *in situ* at Cooper's Brewery an increase in pH value of the filtrate of 2.0 pH units was observed. For large grade zeolite-A the pH increase of the filtrate was less than 0.5 pH units. This increased pH of the filtrate with both grades of zeolite-A was demonstrated to be due to the release of sodium ions from the filter medium. Additional experiments were conducted to exhaust the sodium from the filter media of both the small and large grade zeolite-A. The pH of the filtrates was monitored for between 8 and 16 h of continuous filtration to determine if all sodium could be exhausted from the medium. A practical constraint was that the filter cake became clogged with yeast and other solid particles from the beer haze before a noticeable change in pH of the filtrate could be observed.

Sensory analyses of filtrates of each of the five selected media were carried out by 16 industry *noses*⁶ to assess: colour, aroma, taste, clarity and *drinkability* (= overall impression). Overall the large grade zeolite-A filtrates compared satisfactorily with the DE filtrates in ratings of differences from the Descriptive Method employed in the brewery industry for colour, taste, aroma, clarity and drinkability.

⁶ Professional noses from within Cooper's Brewery Ltd, Leabrook, SA 5068.

Small grade zeolite-A filtrates however compared poorly where the filtrate was regarded as inferior to DE, filter sand and silica, by all the members of the panel of noses. Small grade zeolite-A was further found to have a significant adverse effect on the filtrate taste using the Triangular Method widely employed commercially for establishing taste. Therefore small grade zeolite-A was deemed an unsuitable filter substitute for DE in the clarification and removal of haze constituents in commercial beer.

Large grade zeolite-A however appears to be a suitable substitute filter medium for DE in the removal of haze constituents in beer. Importantly it can be readily substituted for DE without the need for significant changes in brewery industry process equipment and protocols for preparation.

The findings from this study are sufficient to strongly recommend a focused study on contributing chemical and mechanical factors to the (small) pH increase of the filtrate using large grade zeolite-A. It is not known whether a range of zeolites might also provide a practical substitute to DE, present understanding must therefore be said to be limited. Other zeolites proposed for the removal of haze from beer would need to be evaluated experimentally. The pilot plant and procedures developed for this study would be readily applicable for such an undertaking. An important justification for future work is that a suitable substitute for DE such as zeolite-A is seen as timely in view of the significant health risks associated with the established carcinogenic properties of DE.

There is no evidence available to show that zeolites have been studied as an alternative to DE for the removal of haze (in beer or wine). Therefore the findings reported in the present study for zeolite-A, together with earlier findings from the polishing of white wine, strongly indicate the basis for development of IP patent(s).

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Many thanks are extended to all in Department of Chemical Engineering, Adelaide University, for the warm and friendly environment. I hope the results of my work justify the confidence and expectations of all and also of my family and friends.

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CHAPTER 1 - INTRODUCTION

Beer is one of the most popular alcoholic beverages in the world and is consumed in large amounts in almost every country. Worldwide production exceeds 1 billion hectolitre a year (Hermia and Brocheton 1993).

Removal of microbiological and non-microbiological particles in solution during the *clarification*¹ process in brewing is essential to achieve a quality, bright beer. Before packaging, the beer undergoes the final stages of cold conditioning, *filtration* and pasteurisation (or sterile filtration). This conditioning stage allows the settling of suspended yeast and other particulate materials that may cause cloudiness or *haze* in the final product. Once the beer has settled, it is cold filtered (0°C) to ensure precipitation of the haze-active *protein* complexes (Burrell and Reed 1994). A clear beer, free from these constituents is much preferred by consumers and is beneficial for the producers by eliminating problems of spoilage.

Diatomite (also known as DE, diatomaceous earth, *kieselguhr*) has been the most frequently used filter aid in beer filtration since the 1920's. However, with increasing cost to breweries of DE, expensive landfill disposal and its recent classification as a health hazard (IDPA 1998 a, b), a substitute filter media with similar filtering capabilities as DE is sought. This background led to a search for an alternative filter media to DE for the brewing industry.

Based on a recent and confidential experimental study (Davey, Kadir and Pecanek 1997) with wine, *zeolite-A*, an aluminosilicate crystalline material, was demonstrated as a potential substitute for DE in the filtration of beer. The crystal size distribution of *zeolite-A* was found to have significant effect on adsorption rates in molecular sieves and its three dimensional cages contain void spaces that can trap cations and other molecules.

The particular *zeolite-A* used is of interest as it can be synthesised from *kaolin* - a naturally occurring clay. *Kaolin* is abundantly present in Australia and *zeolite-A* is a type that has

¹ see Appendix A for a definition of terms used throughout.

been produced in the Department of Chemical Engineering, Adelaide University as a potential "value-add" step to vast kaolin deposits owned by a client of Adelaide University (Anon. 1996 a; Davey and Daughtry 1997).

The principal objectives of this study are to:

- determine the effectiveness of zeolite-A in removing the haze-forming components in beer
- examine the effect of zeolite-A on the properties of the beer *filtrate*
- compare the filtering capabilities of zeolite-A with those of DE based on filtration characteristics and selected microbial, physical and sensory analyses of the filtrate
- assess the practical suitability of the synthetic zeolite-A as a filter medium for beer.

A summary of the relevant literature is presented in Chapter 2. This chapter includes a brief introduction to beer filtration and focuses on the relevant physical characteristics of DE and zeolite-A. It highlights the fact that zeolites have not been experimentally evaluated for filtration of beer. Published studies of the filtration of white wine using zeolite-A are assessed for usefulness for the filtration of beer.

Chapter 3 outlines the methodology, selection of materials and preparation for the experimental studies. The synthesis and construction of a special pilot plant is presented. Safe operating procedures (*SOP's*) for this test pilot plant are highlighted and details are presented as a special appendix (Appendix B).

In Chapter 4, a comparison of the filtration characteristics obtained in the pilot plant for zeolite-A and those of DE together with filter sand and silica is presented. By using a number of additional filter media the effect of possible bias is reduced in differentiating between DE and zeolite-A in a wide spread of results.

Two grades of a commercially available zeolite-A are used. These are small grade (particle size diameter between 63-125 μm) and large grade (particle size diameter between 125-250 μm). These particle sizes cover the size range of DE particles widely used in the beer industry of 60 to 100 μm diameter.

Following initial experimental studies in the laboratory, all experimental testing was carried out *in situ* in a commercial brewery. In this way, commercial protocols, preparation and experimental filter assessments gave a realistic and practical simulation of the filtration of haze from beer using zeolite-A.

Additional results obtained from sensory analyses of the consequent filtrates by industry *noses* are also presented. Performance of the selected filter media are highlighted, compared and discussed.

Chapter 5 presents a summary of the study together with conclusions and suggestions for further work.

The important terms used throughout are defined in Appendix A and all Notation used is listed at the back of this thesis.

CHAPTER 2 - LITERATURE REVIEW

2.1 Introduction

Filtration is an important unit process in a brewery. It involves the removal of microbiological (*yeast*) and non-microbiological (protein complexes and other filtration auxiliary particles) components. Failure to remove these components can affect the appearance (clarity and brightness), shelf life of the resulting beer product and its attractiveness to consumers.

Diatomaceous earth (DE) has been very widely used since the early 1920's as filter media. Its human carcinogenic classification by the International Agency for Research on Cancer (1997) however has alarmed brewers and all concerned with health hazards in its handling. DE is a white powdery substance that is first prepared as slurry in water and then made into a filter cake for filtration. In its wet state it is not regarded as carcinogenic.

In a search for a substitute for DE in the filtering (*polishing*) of white wine, Davey, Kadir and Pecanek (1997) showed that a synthetic zeolite-A gave similar performance. The zeolite they studied was zeolite-A that had a similar appearance to DE in its dry form. Zeolite-A can be prepared in an identical manner as DE and would therefore fit in with the protocols used in large-scale, commercial filtration. This is seen as a major advantage.

Against the background of world production of 1 billion hectoliters a year (Hermia and Brocheton 1993), and health hazards of the current DE filter media, evaluation of zeolite-A as a substitute filter media was undertaken for the filtration of beer.

2.2 Beer Haze, Clarification and Filtration of Beer

Clarification of beer is done to remove constituents such as yeast, protein complexes and other small particles that cause cloudiness. Potential spoilage microorganisms when not removed, or kept at minimal level, can cause not only hazy beer but can also post economic losses due to a reduced product shelf-life and inconsistency of product quality.

A typical brewing process is presented schematically as Figure 2.1. Unit operations shown as numbers 1 to 6 illustrate the preparation of the *wort* for fermentation and the addition of yeast. Of particular interest however is the unit operation shown as number 7, Filtration.

After *fermentation*, the resulting beer is cloudy and has to undergo a clarification stage to obtain a *bright*, clear product before it is packaged (into kegs, bottles or cans). Combination of methods such as sedimentation, use of *finings*, centrifugation and filtration can be used to achieve satisfactory clarity of product.

Sedimentation relies on gravity. Suspended particles that are denser than water settle at the bottom of the tank, but not all the unwanted sediments may be removed by this method. Other clarification processes might be needed, such as the addition of finings.

The addition of finings involves adsorption, chemical reaction and possibly physical movement. Proteins and yeast are adsorbed on fining agents (such as bentonite, gelatine, casein, isinglass, albumin, egg white, nylon and polyvinyl pyrrolidone (PVPP)) to create larger particles from smaller ones. These can then be removed from the solution by using a *centrifuge*.

Centrifugation requires careful control to avoid undue oxidation and loss of alcohol during the process (McCabe and Smith 1976). It uses centripetal force to push the more dense solid particles to the bottom of the container. This can be used as the primary clarification step in the removal of larger particles and of yeast.

Particles causing haze can also be removed from the liquid by passing through a porous or fibrous material. This unit operation step is called filtration. There are many types of filtration processes and more than one mechanism may play a part. The filter medium and bed depth are suitable only for the removal of small quantities of solids because of low loading of solids. As the particles become deposited in the filter, the retention becomes greater in the upstream side of the medium, leading to blockage. Usually, blockage of the filter is avoided by using different size ranges of the media, the finest being at the downstream side (Ward 1997).

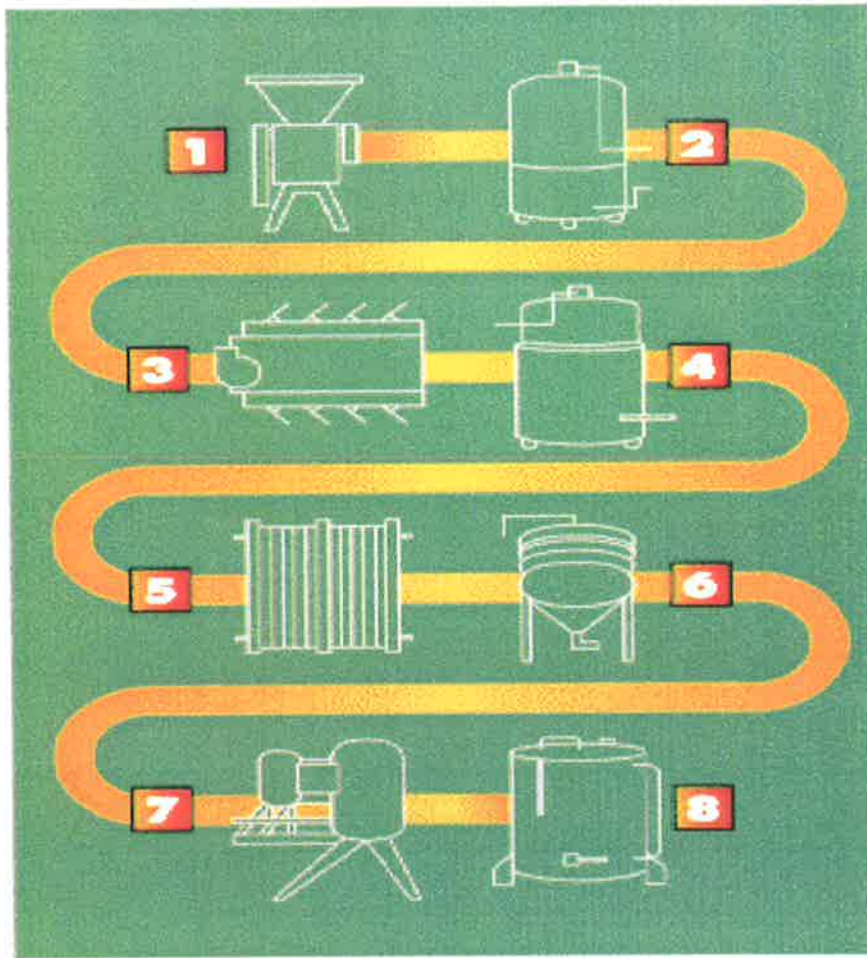


Figure 2.1 A typical brewing process

Map Legend

1	milling	malt barley is cracked
2	"combi" vessel	masher/lauter vessel
3	manifold	control station for liquid transfer
4	brew kettle	wort is brought to boil and mixed with hops
5	plate heat exchanger	cools wort to correct fermentation temperature
6	fermenter	yeast is added for distinctive composition and flavour
7	filter	yeast is removed leaving a brilliantly clear beer
8	serving vessel	receiving vessel

The most commonly used filtration unit operation is the Cake Filtration Method. The permeability or resistance of the *cake* is the most important factor in cake filtration. This can be controlled by altering the particle size distribution of materials, or even adding other solids. The method is most often used in combination with other filtration methods.

Clogging of the pores of the filter medium is reported as the main problem in filtration of beer. Clogging reduces the filter life. The pore size of some filters is sufficiently small to remove yeast cells of diameter of 2-7 μm (Hermia and Brocheton 1993) and most bacterial cells, but filters with 0.8-1.0 μm diameter can be used in the removal of the microbial components to prevent clogging. Yeast cells and complex colloidal materials such as gums (glucans and pentosans) that are derived from the cereal grains and carried through the brewing process can cause clogging. To avoid clogging, breweries use *body-feed* filter aid, the principle of which is to build up the filter cake on a solid support *pre-coated* with the filter media. The unfiltered liquid is dosed with the media, building up the bed as beer is filtered.

2.3.1 Diatomaceous Earth (DE)

2.3.1 DE and other Important Names

The traditional filtration media is diatomaceous earth (DE). DE is used to aggregate and collect the suspended solids. Diatomaceous earth also known as *kieselguhr*, diatomaceous silica or diatomite (Perry and Green 1997) is a naturally occurring light-colored, porous, sedimentary rock that is composed of fossil diatoms, microscopic single-celled aquatic plants. It is almost pure silica or silicon dioxide, namely, $\text{SiO}_2 \cdot n\text{H}_2\text{O}$. It is chemically inert. DE is commonly used as a filter medium for wines, beers and swimming pool filters.

2.3.2 Chemical Makeup

The frustules (cell walls) of the diatoms are made up of silica and contain many fine pores. The fine frustules make it an excellent filtering material for beverages, chemicals, industrial oils, cooking oils, sugar, water supplies, varnishes, lacquers, jet fuels and antibiotics. Its low abrasive properties make DE suitable for use in toothpaste, non-

abrasive cleansers, polishes and buffing compounds. It is relatively inert, with high absorptive capacity, large surface area, low bulk density and relatively low abrasion.

A scanning electron micrograph, presented as Figure 2.2, shows clearly the diatoms of DE.

DE consists of 90% silica with the remainder of aluminum and iron oxides. Commercially available DE can be natural, non-flux (straight) calcined or *flux*-calcined.

The natural diatomite is generally white in color, which is almost entirely amorphous, and may contain small amounts of crystalline silica in the form of quartz. The straight calcined product is calcined at high temperature wherein the organics and volatiles are removed and the colour typically changes from off-white to tan or pink. Calcining greatly increases the amount of crystalline product by the conversion of amorphous silica to crystalline silica during calcination. The crystalline silica produced is mostly cristobalite. Flux calcinations greatly increase the proportion of cristobalite produced.

The cristobalite is capable of causing silicosis when large amounts of it are inhaled. Silicosis is a fibrotic lung disease that has been associated historically with the inhalation of silica-containing dusts (Ricci Bros. 1999). The effect of silicosis on lungs is shown in the photograph presented as Figure 2.3. This shows the manifestations of the disease that includes the development of scar tissue in the lungs that can be progressive and disabling and can lead to death.

2.3.3 Carcinogen Classification

In 1987 the classification of DE was category 2A, a "probable carcinogen to humans" as classified by the International Agency for Research on Cancer (IARC), a WHO agency. Significantly, in 1997 DE was re-classified to "human carcinogen" category 1 by the IARC (IDPA, 1998 a, b).

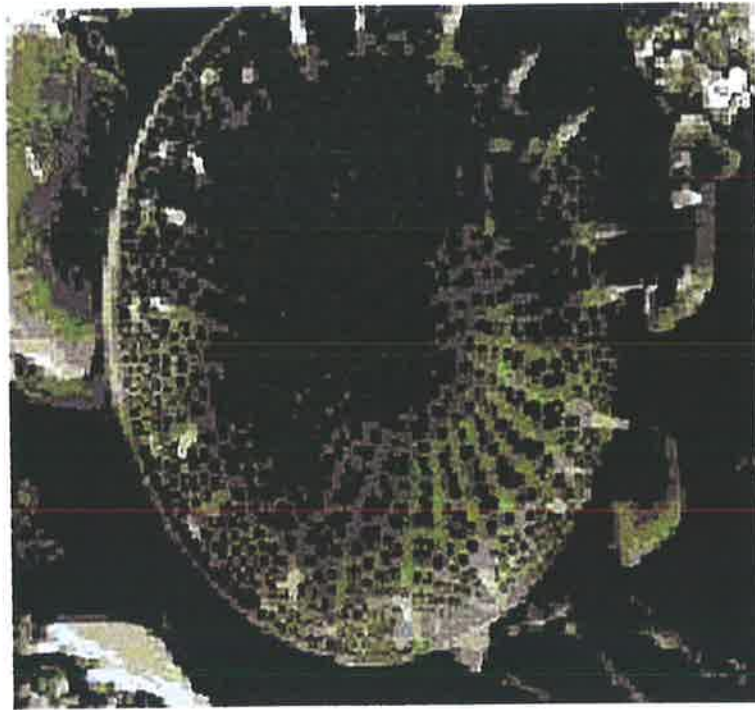


Figure 2.2 Scanning electron micrographs of diatoms
(*adapted from* Hunt and Nutt 1999)

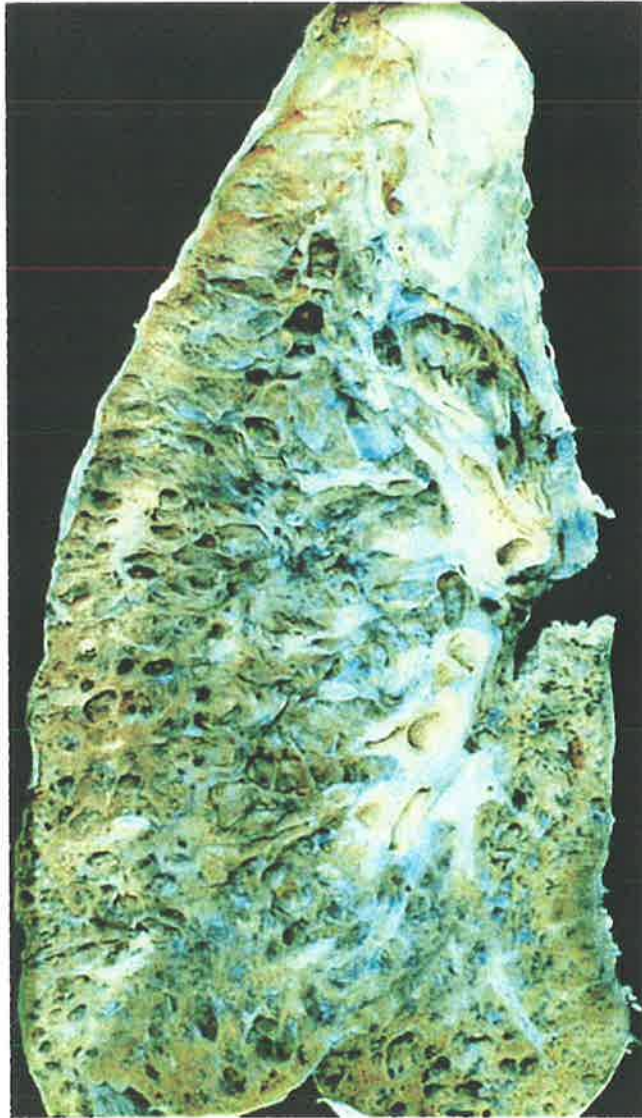


Figure 2.3 Lung affected by silicosis
(*adapted from* Hunt and Nutt 1999)

2.3.4 Quantities Used World Wide

DE is imported into Australia by Filchem Pty Ltd. The demand for DE² in Australia is about 12,000 tonne per annum, with 1,500 tonne used in SA each year. An estimate of the global demand for DE is about 50,000,000 tonne per annum (Anon. 1996 b). This encompasses both the wine and brewing industries.

Filtration using DE as the filter medium produces beer with hazes below 0.6 *EBC* (European Brewing Convention) (Gan *et al* 1997; *pers. comm.* Dr Tim Cooper) and five (5) yeast cells per 100 mL (Hermia and Brocheton 1993). Table 2.1 presents a range and nominal values of physical parameters of interest for DE.

2.4 Zeolite

In contrast to the diatoms of DE, zeolites are hydrated aluminosilicates of the alkali earth metals (Na, K, Mg and Ca) that are found in rocks of volcanic origin. Zeolites can be synthesised from the natural clay such as kaolin, which is abundant in Australia (Anon. 1996a; Davey and Daughtry 1997).

The synthesis of zeolite from kaolin is illustrated in Figure 2.4. The term zeolite-A is commonly used to describe any zeolite having a structural composition of $\{(AlO_2)_{12}(SiO_2)_{12}\}$. Zeolite-A is the most common product of this synthesis as this structure has the most favourable chemical kinetics. The zeolite-A structure consists of relatively small cages. Zeolite-A has SiO_2/Al_2O_3 molar ratio of 2:1.

Zeolites have three-dimensional structure with the silicon and aluminium atoms tetrahedrally coordinated with each other through shared oxygen atoms. The framework has void spaces that can host water and other molecules. These are illustrated in Figure 2.5. The structure of zeolite is illustrated in Figure 2.6.

² The capital cost of the DE mix widely used in the brewing industry is about AUS\$1,110 tonne⁻¹ this compares with that for zeolite-A of about AUS\$1,050 tonne⁻¹ as of January 2000 (*pers. comm.* Dr K R Davey).

Table 2.1 Nominal physical properties of DE

Bulk Dry Density (kg/m ³)	150 – 210
Bulk Wet Density (kg/m ³)	320 – 440
Particle Specific Gravity	2.0 – 2.2
Melting Point (°C)	1710 (softens near 1430)
Boiling Point (°C)	2230
Surface Area (m ² /g)	2 – 30
Moisture (wt.%)	~ 1.0
pH (10% wt/wt slurry)	7.0 – 10.0
Internal Porosity	N/A
Bed Porosity	>95%
Colour	off-white to pink

(Adapted from Perry and Green 1997)

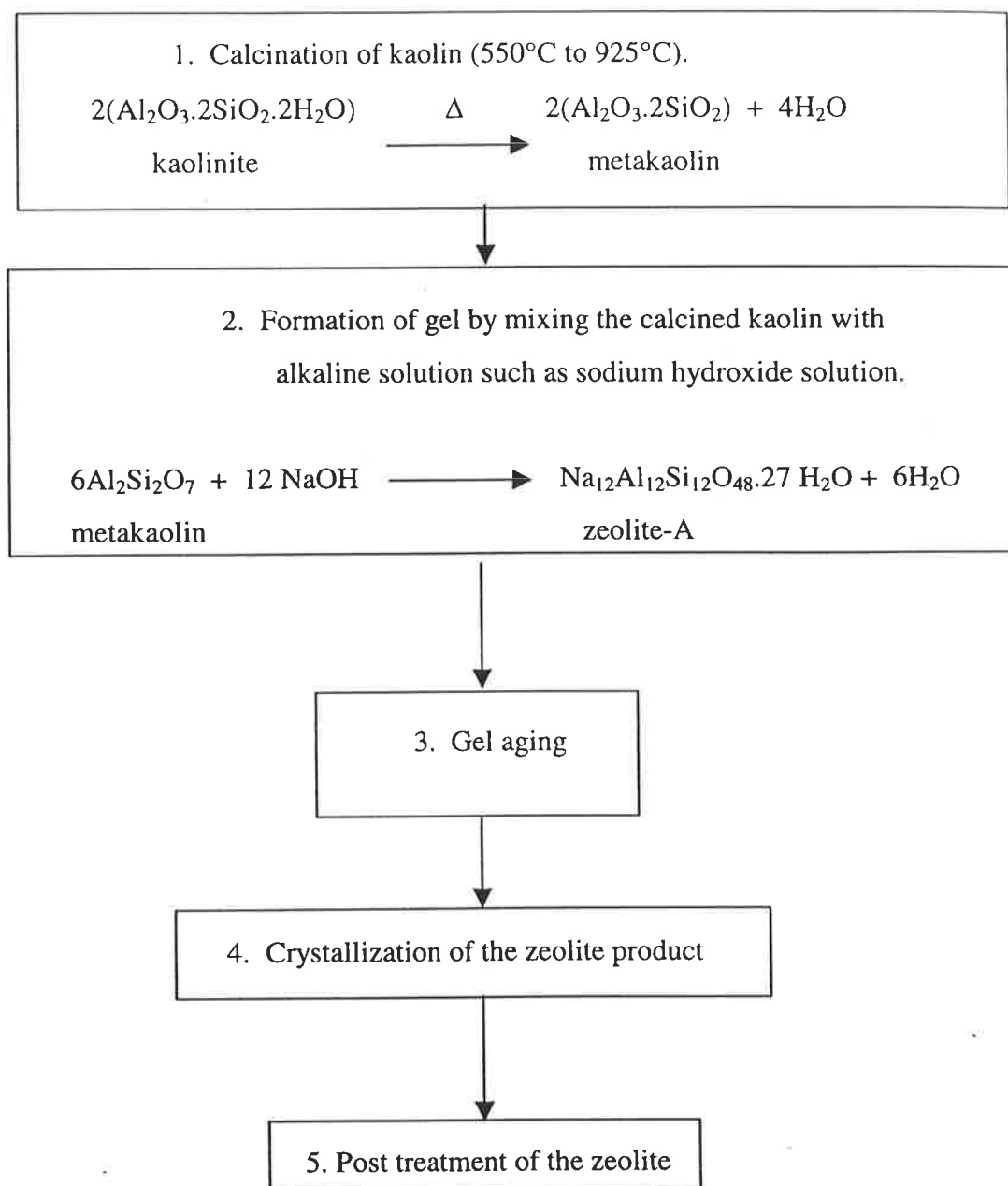


Figure 2.4 Schematic diagram of the synthesis of zeolite from kaolin
(adapted from Anon. 1996a)

The largest volumes of zeolite used commercially are in the detergent formulations where phosphates are replaced as softening agents (Zeolyst International 1999). This is done by exchanging the sodium in zeolite for the calcium and magnesium ions present in the washing water to prevent precipitation of surfactants.

Aside from ion-exchange properties, zeolites can also be used as catalysts and molecular sieves. Zeolite can act as shape selective catalyst by transition state selectivity or by exclusion of competing reactants on the basis of molecular diameter. Its industrial applications include petroleum refining, synthetic fuel production and petrochemical production (Zeolyst International 1999).

The molecular sieve properties of zeolites can be exploited in drying, purification and separation technology. This sieve property is due to its unique structure where 99% of adsorption occurs on the internal surface of the zeolite, but components must physically pass through the desiccant pore openings to be adsorbed (Grace 1999). The different sizes and shapes of the channels of the different zeolites allow them to be used as selective molecular sieves.

Naturally occurring zeolites may contain small percentages of crystalline silica, whilst the synthetically produced may not contain any at all, depending on the starting material and the conversion process used in manufacture.

A range and nominal value of physical parameters describing zeolite is given in Table 2.2. These are generally comparable to DE (Table 2.1). Both materials have similar appearance, moisture content and pH value (of 10% wt/wt slurry). However, zeolites have significantly larger surface area per unit gram, 600 to 700 m²g⁻¹ compared to 2 to 30 m²g⁻¹ of DE. Although the particle specific gravity of both is very similar, zeolites have nearly twice the density of DE both dry and wet.

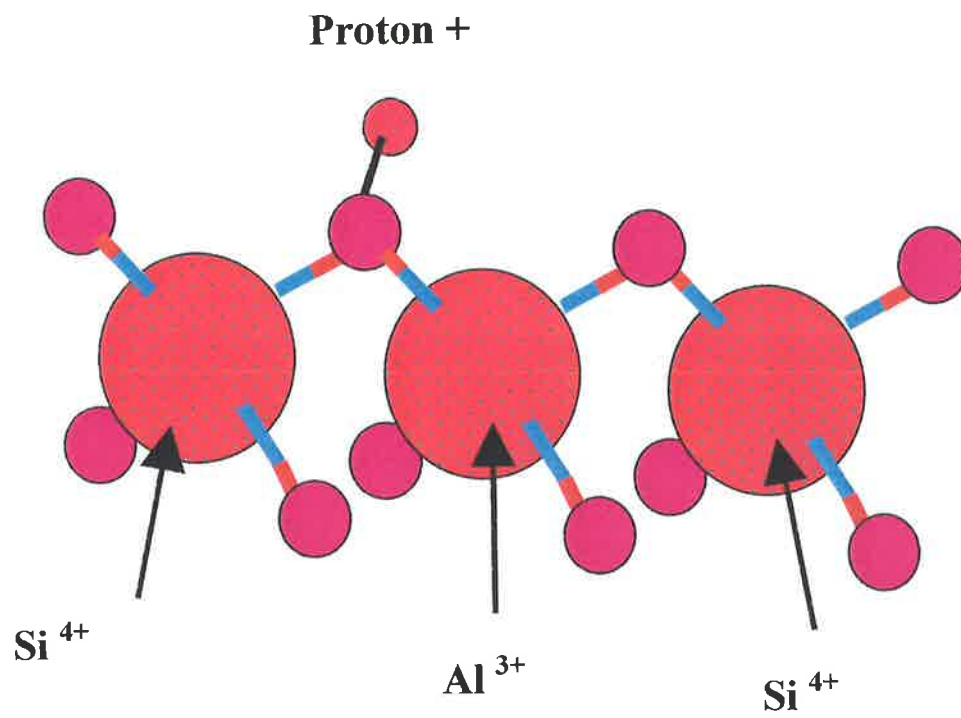


Figure 2.5 Tetrahedral co-ordination in zeolite structure
(*adapted from Trent 1992*)

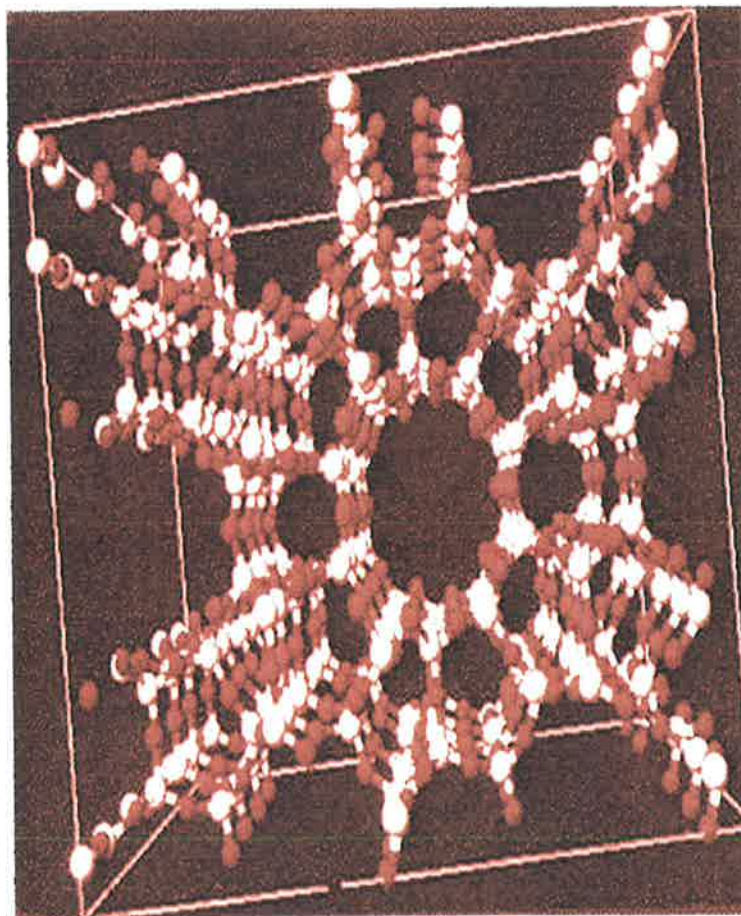


Figure 2.6 A model of a zeolite structure

(sourced from Cache Scientific, Inc./Encyclopedia Britannica 1999-2000)

Home Page <http://www.eb.com>

Table 2.2 Nominal physical properties of zeolites

Bulk Dry Density (kg/m ³)	680 – 760
Bulk Wet Density (kg/m ³)	850 – 950
Particle Specific Gravity	2.2 – 2.8
Melting Point (°C)	> 1600
Boiling Point (°C)	unknown
Surface Area (m ² /g)	depends on PSD and treatment; typically 600 – 700
Moisture (wt%)	~ 1.5
pH (10% wt/wt slurry)	7.5
Internal Porosity	30 – 40%
Colour	off-white to tan

(Adapted from Perry and Green 1997)

2.5 A Mathematical Model for Filtration

Flow through a packed bed of filter media can be explained by Darcy's basic equation which relates the flow rate V of filtrate with viscosity μ through a bed of thickness L and cross sectional area A , to the driving pressure ΔP (Nock 1997):

$$V = \frac{K A \Delta P}{\mu L} \quad (2.1)$$

K is the constant for the permeability of the filter bed, the reciprocal of which is defined as the *cake (bed) resistance*, R .

Two other important properties that define a filter medium are the porosity ε , defined as the fraction of volume of the bed not occupied by solid material (also known as bed voidage) and the specific resistance, α . Respectively, these are defined as:

$$\varepsilon = 1 - (\rho_B / \rho_P) \quad (2.2)$$

$$\alpha = \Delta P / \mu u_w \quad (2.3)$$

2.6 Analysis and Summary of the Pioneering Work with White Wine

The relatively recent and confidential study of Davey, Kadir and Pecanek (1997) pioneered the application and use of zeolite as a substitute filter medium for wine. This research was financed through Adelaide University's commercial office, *Luminis* Pty Ltd, Pulteney Street, Adelaide. There are no other published reports of the use and findings of zeolite-A as a substitute filter medium for the removal of haze in either wine or beer (*see* Section 2.7)

An assessment of six alternative filter media was experimentally evaluated as possible replacements for DE in the polishing of white wines. White wines were selected for assessment because clarity (haze or turbidity) is more important than with red wines.

The Australian Wine Research Institute, Adelaide, independently carried out chemical analyses of the wine and resulting filtrates. These included pH, sulphur dioxide, alcohol, metals (Cu, Fe, K, Na and Ca), heat stability and turbidity.

Oenological tests (taint, colour, taste) were undertaken by a selected panel of wine makers at Southcorp Wines Pty Ltd, Nuriootpa, SA, using coded samples.

The protocols for preparation of the filter media simulated conditions and practices currently in use with DE in the wine industry. A DE typical of that widely used in the wine industry was the control filter medium. Three particle sizes of a commercial zeolite-A, respectively, 125-225 μm , 5 μm and 62-125 μm , were used. These particle sizes were selected to adequately cover the range of the particle size of DE used commercially of 60-100 μm .

The important findings included that:

- zeolite-A resulted in fluxes very similar, or greater than that of the control DE for the range of particle sizes
- oenological testing ranked zeolite-A similarly to DE.

2.6.1 Safe Handling Issues

Because the filtrate from either white wine, or beer, was for human testing (oenological evaluation) and consumption, it was of a major health significance. Davey, Kadir and Pecanek (1997) had established safe handling issues through extensive consultations with Dr. Allan Pring, Curator of Minerals and Meteorites, Division of Natural Science, Museum of SA, Adelaide. Dr Pring is an acknowledged world expert on zeolites.

A summary of the examination of safety issues is appended as Appendix D.

In the following Chapter 3, the synthesis of a special pilot plant to assess the filtration characteristics of zeolite-A against those of a commercial grade zeolite-A is presented. The particular commercial zeolite-A selected is that judged as nearly the same as the zeolite-A manufactured within the Department of Chemical Engineering, Adelaide University (Anon. 1996a; Davey and Daughtry 1997) from naturally occurring deposits of kaolin. To assist replication of the study, the behaviour of the particulate bed during start-up is discussed based on a protocol established in the course of the work.

2.7 Concluding Remarks

Based on the pioneering work of Davey, Kadir and Pecanek (1997), it is clear that zeolite-A might be a useful substitute for DE in the reduction of haze in beer.

No reports of zeolites used as filter medium for either wines or beer appear in the published literature. This finding is reinforced by the results of a worldwide search of the patent literature carried out as a consultancy to Adelaide University by APT Patent and Trade Mark Attorneys, Adelaide SA 5000, in April 2000. They concluded that " There are no findings which disclose the use of zeolite for haze stabilisation in wine or beer. Accordingly, (this) search has not retrieved any entries that foreshadow difficulties in achieving some patent right".

Against the background of the carcinogenic classification of DE and the successful trials with zeolite-A as a polishing agent for white wine and the potential for development of IP rights, a study of the filter capabilities of zeolite-A for removing haze from beer is both opportune and timely.

CHAPTER 3 - MATERIALS AND METHODOLOGY

3.1 Introduction

Evaluation of zeolite-A as a potential alternative to diatomaceous earth (DE) as a filtering media for removal of haze in beer involved a synthesis, design and construction of a pilot plant to simulate industrial conditions. To best accommodate realistic studies (and satisfy Australian Customs and Excise Services), experimental studies were carried out *in situ* in Coopers Brewery Ltd, Leabrook, SA 5068. The quality of filtrate samples was then conveniently managed and evaluated and directly compared against routine commercial beer produced by conventional DE process methods.

Consequently, the experimental program focused on the:

- design and location of an adequate pilot plant
- pilot plant start up
- safe operating procedures of the plant
- preliminary tests and establishment of experimental protocols
- sampling and management of samples for microbiological and sensory analyses.

At the outset it was found that a more controlled and reproducible measure of the filter bed dimensions (filter depth) was obtained with carefully determined masses of each of the filter media, rather than attempting to reproduce a fixed bed depth. Masses of either 35 or 65 g were used in preliminary studies carried out within the laboratories of Adelaide University (departments of Chemical Engineering and Microbiology and Immunology) and masses of 3.63, 11.23 and 18.83 g, respectively, when *in situ* at Cooper's Brewery Ltd.

These masses simulated the depth of the filter bed used in the commercial production of beer.

All filtrates that were used for microbial and sensory analyses were standardized. This was done by using a fixed bed mass of 18.83 g of medium and a fixed pressure driving force of 180 kPa.

3.2 The Pilot Plant

3.2.1 Design

An experimental pilot plant based around an *egg pump* fulfilled necessary conditions. In this, pressure of a gas, in a leak-proof vessel, is increased above the liquid which is forced out and through related pipe-work. This has three advantages over a conventional pump:

- no food-grade pump is required
- increased control of bed pressure drop
- ease of cleaning.

A food grade nitrogen gas (BOC No. 036) was selected for the experimental studies. All wetted surfaces were food-grade 316 stainless steel. This was important as experienced noses in part consumed the filtrates during assessment.

A schematic flow diagram of the pilot plant is presented as Figure 3.1. A photograph of the actual pilot plant *in situ* at the *Lager Cellar* (Figure 3.2) of Cooper's Brewery Ltd, Leabrook, SA 5068 is shown in Figure 3.3.

The plate and frame Seitz filter routinely used in the brewery for haze removal is illustrated as Figure 3.4. The *filtrate* from this filter was used as a "control" throughout the experimental program.

A definition of instrumentation labels for the experimental pilot plant is shown schematically in Figure 3.1 is as follows:

- V1** food grade nitrogen supply valve
- P1** indicates pressure of food grade nitrogen supply from the gas cylinder
- V2** pressure regulator to control the pressure supplied to the pilot plant downstream
- P2** indicates pressure of nitrogen supply after going through pressure regulator V2
- V3** control regulator to set pressure of nitrogen supply to the pressure vessel -
V3 keeps the pressure constant even if the upstream pressure fluctuates
- V4** three-way valve allows manual venting of the process line and pressure vessel
- P3** indicates the pressure in the pressure vessel
- V5** set to vent at 6.0 bar - prevents the pressure in the vessel from becoming too great as to be unsafe
- V6** emergency shut off - can be used to instantaneously stop the flow from the filter vessel
- P4** indicates pressure just above the filter cake.

The pilot plant was designed such that when there was an excess pressure from the nitrogen tank, the tank could be vented from the line to the atmosphere manually using V3 and thereby preventing any damage to V4. The pressure regulator could be set for accurate regulation (between 0 to 1000 kPa). The pressure was monitored using the pressure gauge fitted to P3. A pressure relief valve (V5) was fitted to the lid of the pressure vessel to prevent over-pressure and was set at 620 kPa. A ball valve (V6) was installed as an emergency shut off.

Details of construction of the filter vessel are given as Figure 3.5.

The filter vessel consisted of a piece of clear polycarbonate tubing clamped together with two stainless steel flanges. The filter media is supported within the polycarbonate tubing by a sintered-stainless-steel plate. A pressure gauge (P4) is fitted above the filter vessel to measure the pressure drop across the filter bed.

3.2.3 Start Up

The DE, silica and zeolite-A filter media were assumed to be *sterile*.

The pilot plant was sterilised *in situ* using a commercial sodium metabisulphite solution (or a 70% v/v ethanol solution) at start up. This included all downstream equipment (filtrate hose, lid and sample container).

The filter sand was not regarded as sterile. The sand medium and all surfaces of the pilot plant were sterilised with the ethanol solution prior to filtration.

Importantly, investigatory samples of beer filtrate from the sintered plate only in place in the pilot plant (i.e. no filter medium) highlighted that no detectable haze was removed by the sintered material.

3.3 Preliminary Tests and Preparation of Filter Media

Preliminary trials with the pilot plant were carried out using different media with tap water, home-brewed beer and a purpose-made, beer simulant (as a yeast solution). DE as Celite 503³, pumice, cotton wool, filter sand, two size grades of zeolite, perlite and silica were trialled.

The pilot plant was initially checked for possible faults by running tap water at a pressure between 206 - 620 kPa (30 - 90 psig). Home-brewed beers (*supplied by* M. Nutt, D. Edwards and R. Ivanovic) were filtered using DE (Celite 503) and a small grade zeolite-A with a particle size of 63-125 μm .

The simulated beer (*see* Appendix C for details) was used to assess each of the filter media. Resulting filtrates were analysed in the Microbiology Laboratory of the School of Medicine, Adelaide University.

³ obtained from Southcorp Wines Pty Ltd, Nuriootpa, SA and marketed by FilChem Pty Ltd VIC.

Zeolite-A is available (*from* Dri-Packs Pty Ltd, NSW) in the form of beads of 3 to 5 mm diameter. These were ground to appropriate size for filtration at the Minerals Processing Laboratory (Mr Keith Quast), University of South Australia, Adelaide, which houses grinding and screening equipment (Figure 3.6).

The zeolite-A was ground to both a size range of 63-125 μm (small diameter) and 125-250 μm (large diameter) using a small rod mill with stainless steel rods. The desired particle sizes were obtained with a continuous vibrating stack of screens.

The range of particle sizes of zeolite-A can therefore be seen to cover the particle size range widely used with DE.

3.3.1 Safe Operating Procedure (*SOP's*)

Safe Operating Procedures (*SOP's*) were developed for the pilot plant. Importantly, these involved the familiarization and evaluation of the experimental pilot plant for filtration. The *SOP's* are presented in Appendix B. This presents in detail the step by step procedure followed as a standard safety method. Safety concerns included the high-pressure hazards and fine-dust respiratory hazards.

To work at pressures that simulate industry practice, transparent sections were constructed from polycarbonate tubing as this material has both high-pressure rating and impact resistance⁴.

Inhalation of the fine particulate of DE and zeolite-A was eliminated (minimized) through the use of a respiratory mask - including protocols for preparation of slurries of the materials. Details of the relevant safety standard are given in AS 1715-1982⁵.

⁴ the same material as used in polycarbonate lenses, helmet visors worn by astronauts during space travel and riot shields of police.

⁵ *Selection, Use and Maintenance of Respiratory Protective Devices*. AS 1715-1982 pp. 5.

3.3.2 Microbial Tests

Homebrewed beers, tap water and prepared yeast solution (beer simulant) were filtered in the pilot plant using zeolite-A and DE. The filtrates were analysed in the Microbiology Department, Adelaide University for viable, and total, cell count.

Total cell count was assessed using a haemocytometer, and the viable cell counting by the Spread Plate Method (Meynell and Meynell 1970) on Savaud's agar media. The detailed microbial analysis is presented in Appendix C.

3.3.3 Experimental Design for Filtration of Beer Samples

To limit and effectively target the number of experimental flux-time studies with the pilot plant on commercial beer a number of experimental designs were evaluated. A summary of the experimental program adopted for the beer haze trials *in situ* at Cooper's Brewery Ltd is shown in Table 3.1.

The table highlights that a total of 324 separate experimental flux-time experiments were carried out. This number of experiments involved 3 x pressure gradients (70, 125 and 180 kPa), 3 x filter bed masses (3.63, 11.23 and 18.83 g of media), x 5 filter media (silica, filter sand, Cooper's Brewery Ltd commercial DE Mix (SuperCel[®] and FilterCel[®]), large grade zeolite-A and small grade zeolite-A plus the control (i.e. sintered plate only of the pilot plant filter) x 3 replicates each of 2 x sample volumes (3.5 L plus 3.5 L) and 1 x beer (Cooper's Brewery Ltd DB).

Cooper's Brewery Ltd DB beer was selected as it is routinely manufactured. As highlighted in Table 3.1 this experimental program entailed some 700 h of continuous experiment. The experimental program adopted was judged practically feasible for testing of the research aims together with sufficient time for analyses and report writing. (Were all initial eight filter media - additionally, perlite, pumice and cellulose - evaluated experimentally each with three pressure gradients, three bed masses and with, say, five beers a total of 3,645 separate experimental trials).

3.4 Filtration of Beer Samples

The routine operating conditions of the Seitz filter in Cooper's Brewery Ltd are shown in Table 3.2. As shown in the table the temperature of the air surrounding this commercial plant is continuously monitored and controlled at 2 to 4 °C. The operation of the pilot plant to simulate the commercial process conditions is given in Table 3.3. The ratio, flow rate/filter surface area, provided by each is almost identical with a mean value for the commercial Seitz filter over the ranges shown of $222 \text{ mLm}^{-2}\text{s}^{-1}$ (cf $224 \text{ mLm}^{-2}\text{s}^{-1}$ for the pilot plant). Plates 3.1 through to 3.5 show the filter media, respectively, FilterCel, SuperCel, small grade zeolite-A, large grade zeolite-A and filter sand.

Initially, to simulate the brewery practice used for all commercial-scale DE filter cake, a precoat, a precoat plus one batch of body-feed (i.e. additional DE), and; a precoat plus two batches of body-feed respectively, was trialled in the pilot plant.

The precoat procedure involves the preparation of a beer-DE slurry that is applied as a thin layer to the filter support and left to "dry" for a short period of time prior to filtration of the main body of beer. This assists establishment of a stable filter cake. This procedure was not however adopted as standard pilot plant operation because it was not readily reproducible, largely through end and side effects on the sintered support plate.

3.5 Analyses of Filtrates

Containers for collection of the filtrate were sterilised using ethanol solution (70 %v/v), purged with nitrogen to (minimize contact with oxygen) and sealed prior to filtrate collection. Filtrate was collected (about 2.5 L each trial) and stored in the Lager cellar cold room at a temperature of 2 to 4 °C prior to analyses.

Analyses of samples were conducted within the Brewery Laboratory where samples were de-gassed immediately prior to analyses. For each sample three properties were measured: haze, colour and pH.

The haze level of samples was measured using a VOS 4000 hazemeter. EBC (European Brewing Convention) units were registered in a digital read-out indicating the ratio of scattered and transmitted light intensities. A haze reading of <1 EBC is commercially considered a bright (i.e. desirable) beer (Gan *et al* 1977; *pers. comm.* Dr Tim Cooper).

Samples for the colour test were filtered using a standard industry glass filter paper (Whatman GF/C). Absorbance readings were taken at 430 nm using a Varian DMS 200 UV Spectrophotometer.

Sample pH was measured at a sample temperature of 20 °C using a standard pH probe.

3.6 Sensory Analyses of Filtrates

Two methods of sensory analysis were used, the Triangular Method and the Descriptive Comparison. These methods are routinely used in the brewing industry and can be relied on (*pers. comm.* Nick Sterenberg, Cooper's Brewery Ltd).

The Triangular Method uses three samples presented simultaneously and requires the subject to choose the "odd" sample. A "no difference" reply is not recorded. This therefore forces a choice from the subject even when the results are not clear.

Beer filtrates from small grade zeolite-A were assessed against the beer filtered in the Seitz filter (i.e. control) using the Triangular Method. Twelve experienced assessors (*noses*) determined if there was a difference apparent in the two beers.

The Descriptive Comparison method evaluates the beer filtrates by describing aroma, color, clarity, taste and *drinkability* and overall impression. Filtrates are presented all at once to assessors. Instructions are as simple (as is possible) and require the subject to rate the intensity of each characteristic on a scale from 0 to 10, 0 being "poor" and 10 being "excellent".

Details of the two methods can be found in Appendix F and from the discussion of Chapter 4.4. The format for the Descriptive Comparison was developed during the course of this study (*pers. comm.* Dr K R Davey).

3.7 Ion-Exchange

Beer was filtered using zeolite-A as filter media. Samples volumes were collected at intervals of time. These were then tested for pH change. Collection of samples continued until the pH of the filtrate appeared to be stabilized.

3.8 Concluding Remarks

The methodology outlined in this chapter and experimentally employed should be sufficient to establish the eligibility and effectiveness of zeolite-A as a substitute filter media for diatomaceous earth in the removal of haze from beer.

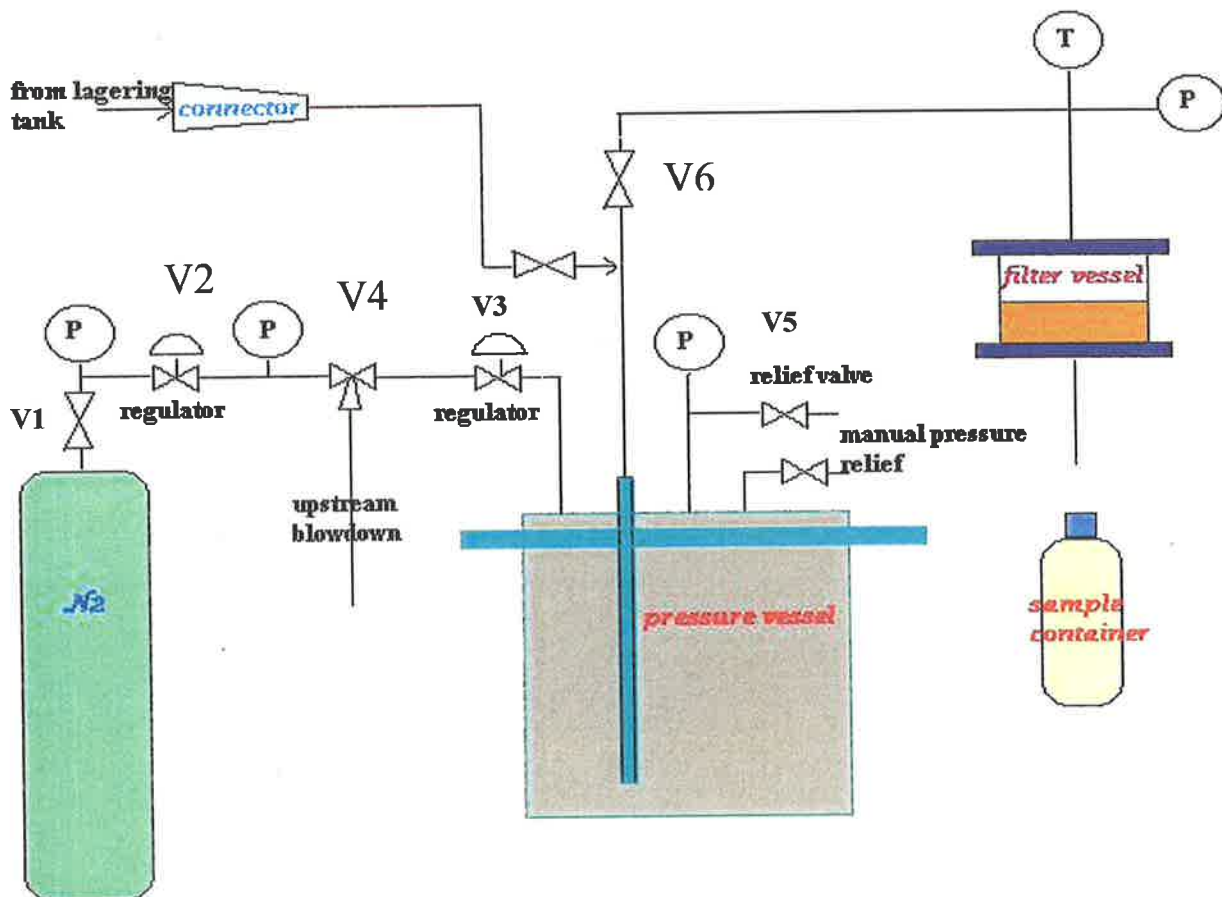


Figure 3.1 Schematic diagram of the pilot plant



Figure 3.2 Lager cellar where beer is kept for maturation
(*courtesy of Coopers Brewery Ltd.*)

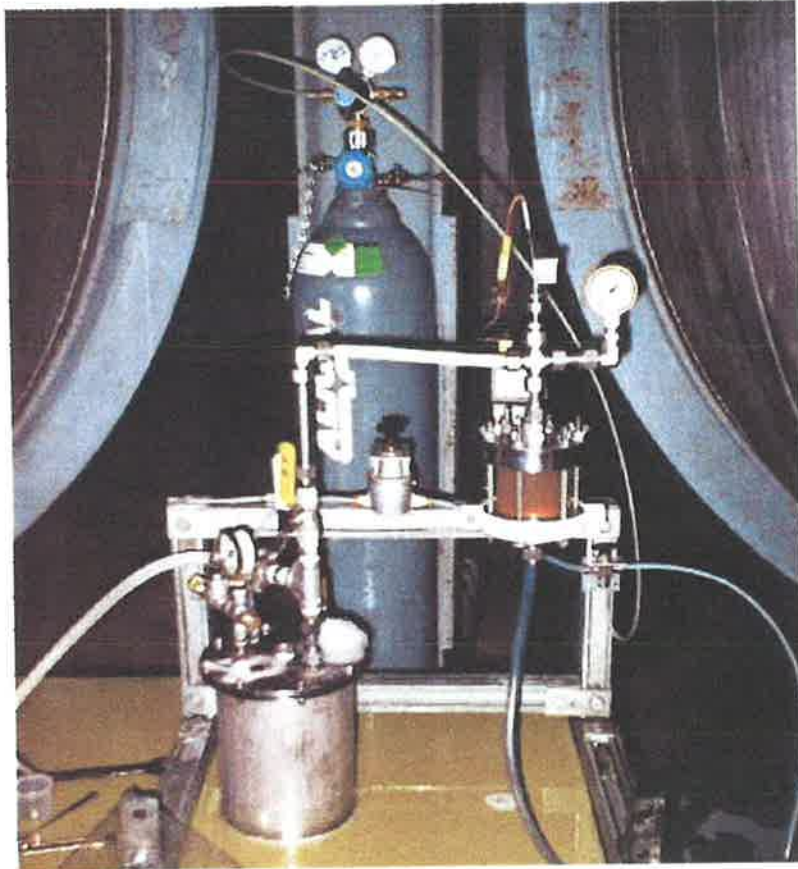


Figure 3.3 Set-up of the pilot plant at the brewery

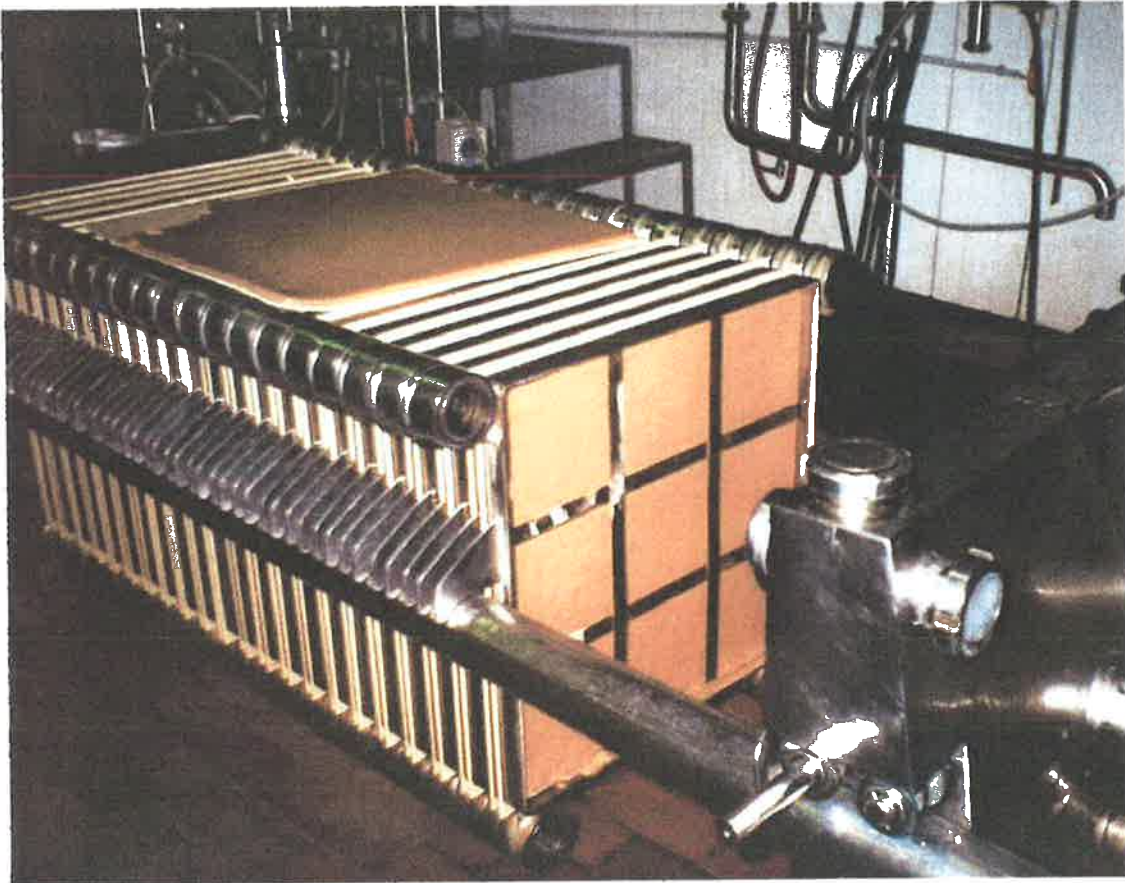


Figure 3.4 Seitz-Werke GMBH 60V plate-and-frame filter
(*courtesy of Coopers Brewery Ltd.*)

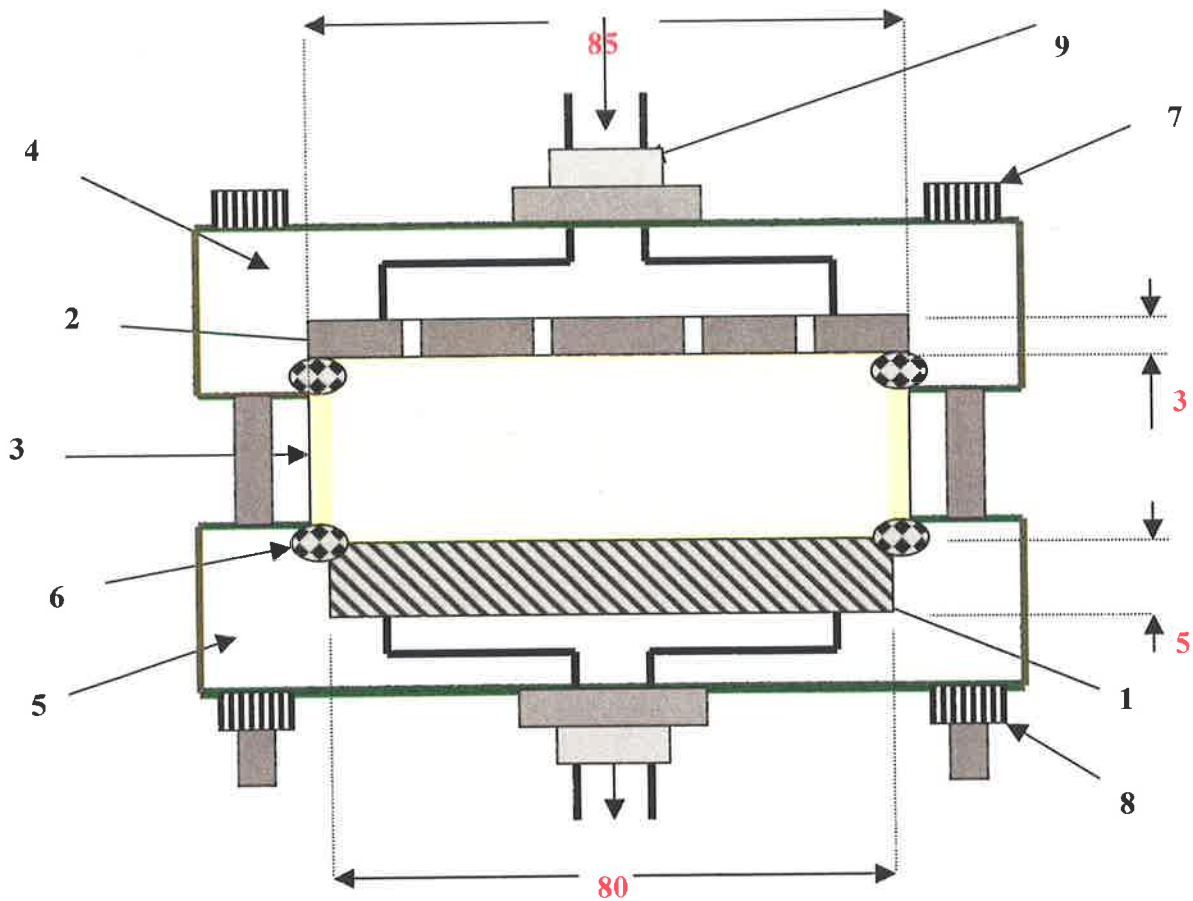


Figure 3.5 Details of the construction of filter vessel

Map Legend

- 1 stainless-steel sintered plate (septum)
- 2 stainless-steel impact plate
- 3 polycarbonate tubing
- 4 upper stainless-steel support
- 5 lower stainless steel support
- 6 O-ring
- 7 bolt
- 8 nut
- 9 half-inch Nylex™ tubing

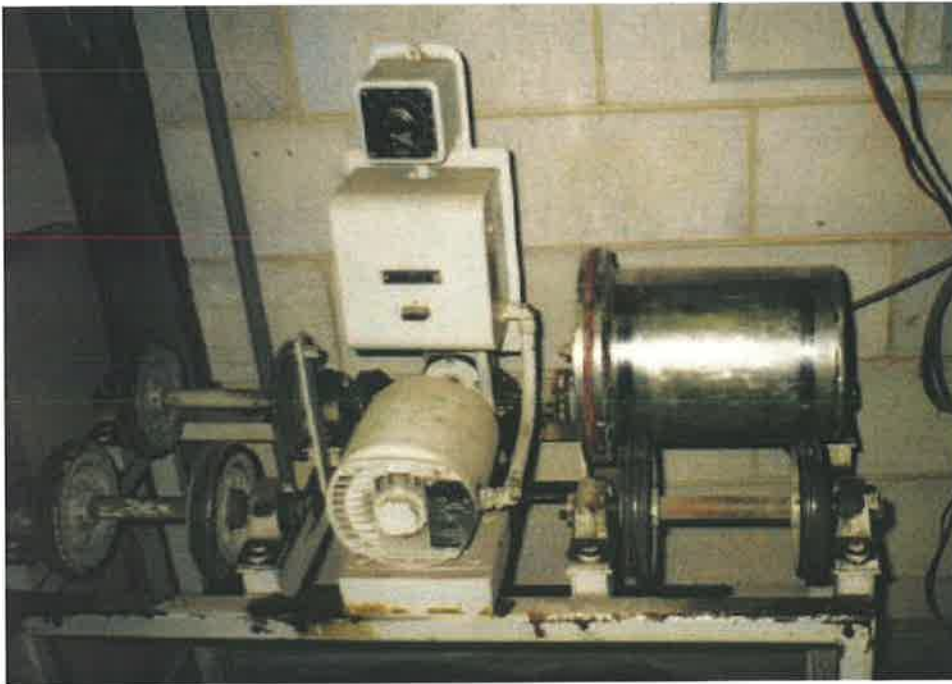


Figure 3.6 The grinding facility at the Mineral Processing Laboratory at the University of South Australia

Table 3.1 Experimental Design

Experimental Design	Number
filter media	5 +1*
pressure drop	3
bed masses	3
beer type(s) (<i>DB</i>)	1
replicates	3
number of samples/replicate	2
<hr/>	
Total Number of Experiments	324
Estimated Time	Number x time (min)
flux-time experiment(s)	162 x 20
sampling run(s)	162 x 180
change-over	324 x 15
cleaning and sterilising	162 x 25
<hr/>	
Total Time Required	41,310 min
	(approx. 700 h)
<hr/>	

* sintered plate only of the pilot plant

Table 3.2 Routine operating data for the Seitz filter at Cooper's Brewery Ltd

Flow rate:	10,000-13,000	[L/hr]
Filter area:	15.03	[m ²]
Pre-coat mass:	11.0	[kg]
Pre-coat loading:	0.732	[kg/m ²]
Body-feed mass:	23.0	[kg]
Body-feed loading:	1.53	[kg/m ²]
Total mass:	34.0	[kg]
Total loading:	2.26	[kg/m ²]
Volume:	23000	[L]
Avg. pressure drop:	159	[kPa]
Temperature of air	-1 to 0	[°C]

Table 3.3 Operating condition for the pilot plant in situ in Lager cellar, Cooper's Brewery Ltd

Flow rate:	3.30-4.29	[L/hr]
Filter area:	4.96×10^{-3}	[m ²]
Pre-coat mass:	3.63×10^{-3}	[kg]
Pre-coat loading:	0.732	[kg/m ²]
Body-feed mass:	7.57×10^{-3}	[kg]
Body-feed loading:	1.53	[kg/m ²]
Total mass:	11.2×10^{-3}	[kg]
Total loading:	2.26	[kg/m ²]
Volume:	7.5	[L]
Pressure drop:	159	[kPa]
Temperature of air:	2 – 4	[°C]



Plate 3.1 FilterCel®



Plate 3.2 SuperCel®



Plate 3.3 Small grade zeolite (63 to 125 μm mean particle diameter)



Plate 3.4 Large grade zeolite (125to 250 μm mean particle diameter)



Plate 3.5 Filter Sand

CHAPTER 4 - RESULTS AND DISCUSSION

4.1 Introduction

Results obtained from the experimental investigation described in Chapter 3 are presented in this chapter. Replicate data are presented for each of five filter media: small and large grade zeolite-A, DE (as Celite 503 in preliminary trials and as Cooper's Brewery Ltd DE Mix⁶ *in situ* in the brewery), filter sand and silica.

Preliminary data obtained from studies within the laboratories of Adelaide University (departments of Chemical Engineering and Microbiology and Immunology) are first reviewed for the initial eight filter media (additionally, cellulose, pumice and perlite) and then data obtained *in situ* at Cooper's Brewery Ltd for each of the five selected filter media. The preliminary trials were carried out with beer simulant (yeast solution), commercially sourced home-brewed beers, and tap water.

The major findings from the experimental studies are summarised comparatively and the adequacy of zeolite-A as a filter substitute for diatomaceous earth in the removal of haze constituents from commercial beer is discussed.

To disguise zeolite-A as a filter medium and to preserve the confidential nature of this study, zeolite-A was referred to as *Ken* in all trials that were carried out away from the laboratories of the University.

4.2 Preliminary Experimental Studies

4.2.1 Microbiological and Bench Tests

Figures 4.1 and 4.2 summarise results of the tap water and yeast solution trials for both DE and small grade zeolite-A. A mass of 35 g of each medium was used. This gave an approximate bed depth of, respectively, 2.5 cm and 1.5 cm. The data are plotted as filtrate

⁶ SuperCel[®] and FilterCel[®]

volume (mL) *versus* time of filtration (s). Values for the bed voidage (ϵ), bed permeability (K) and bed specific resistance (α) were calculated from equations 2.1 to 2.3 for each of the filter media. Figure 4.3 shows the filtrate volume *versus* time for the sintered plate only (that is, no filter medium) from which the bed resistance, R , of the pilot plant was calculated as $R = 11.0 \times 10^9 \text{ m}^{-1}$.

Two commercially sourced home-brewed beers, *Black Rock Lager* and *Dark Ale*, were filtered to remove haze constituents. *Black Rock Lager* was filtered using DE. *Dark Ale* was filtered using both DE and small grade zeolite-A (63-125 μm particle diameter) filter media.

The *Black Rock Lager* filtrate obtained with DE was bright-clear. Total solids were reduced by 28% (Table 4.1).

A direct comparison of the filtering capabilities of DE and zeolite-A was made with the *Dark Ale* beer. Both filtrates showed a satisfactory clear beer. Zeolite-A was more effective in reducing the amount of solids in the filtrate as compared to DE, reducing the total solids by, respectively, 9% and 3.6% (Table 4.2 and Table 4.3). Microbial analyses of both control and filtrate (in triplicate) samples showed the viable yeast cell numbers for both filtered beers was reduced (Table 4.4). The beer samples filtered using DE were reduced from viable numbers of $10^6 \text{ cells mL}^{-1}$ to $10^5 \text{ cells mL}^{-1}$ for both types of beer. For the zeolite-A-filtered beer there was no growth evident from plating the filtrate. This indicates a total removal of all viable cells of yeast.

To evaluate a range of filter media, a yeast solution (*see* Appendix C) was prepared as a test liquid and beer simulant. This liquid was filtered at 206.84 kPa (30 psig) using either 30 g or 35 g of each of the eight filter media. Media included: cotton wool, pumice, perlite, silica, filter sand, DE and the two grades of zeolite-A (small and large).

Results (Table 4.5) showed that DE and small grade zeolite-A were the most effective in filtering out the yeast cells. Cellulose (as cotton wool), pumice and perlite were rejected as unsuitable for further experimental trials in the filtration of yeast cells (haze) from beer. This was because this filter media did not remove an adequate amount of the haze. Further,

Table 4.5 shows that the standard deviation on three replicate filtrations for these three media gave a very large standard deviation of nearly an equal order of magnitude as the mean value. The implication is that the mean pore size varied greatly despite careful experimental technique with each preparation of the filtration bed from these three media.

4.2.2 Flux-Time Experiments

Commercial beer samples were filtered in preliminary trials *in situ* at Cooper's Brewery Ltd using each of the five selected media (silica, filter sand, DE and the two grades of zeolite-A) with three selected pressure gradients.

These were, respectively, 70, 125 and 180 kPa. Three filter beds of each medium were used. The mass of each of these was, respectively, 3.63, 11.23 and 18.83 g.

Figures 4.4 and 4.5 show sample results of the nine trials that were carried out for each of the five media. These are for a pressure gradient of 180 kPa and 3.63 g of filter media and 180 kPa and 18.83 g of each filter media, namely, zeolite-A⁷ small grade, zeolite-A large grade, DE and silica. Respectively, this gave fluxes of: 22, 290 and 390 mLm⁻²s⁻¹, for a bed mass of 18.83 g and a pressure driving force of 180 kPa (Figure 4.4).

The deeper bed of media at the pressure driving force of 180 kPa gave the best flux-time result for each of the five media. This combination had also resulted in the best results for microbial analyses of the filtrate. A pressure gradient of 180 kPa is about 20 kPa greater than the pressure gradient used generally in commercial filtration of beer haze with DE. It is nevertheless a pressure gradient that could readily be used routinely with existing commercial equipment and preparation protocols.

⁷ **Important Note:** for the Brewery trials zeolite-A was referred to as *Ken* to disguise it as a medium and to preserve the confidential aspects of this study.

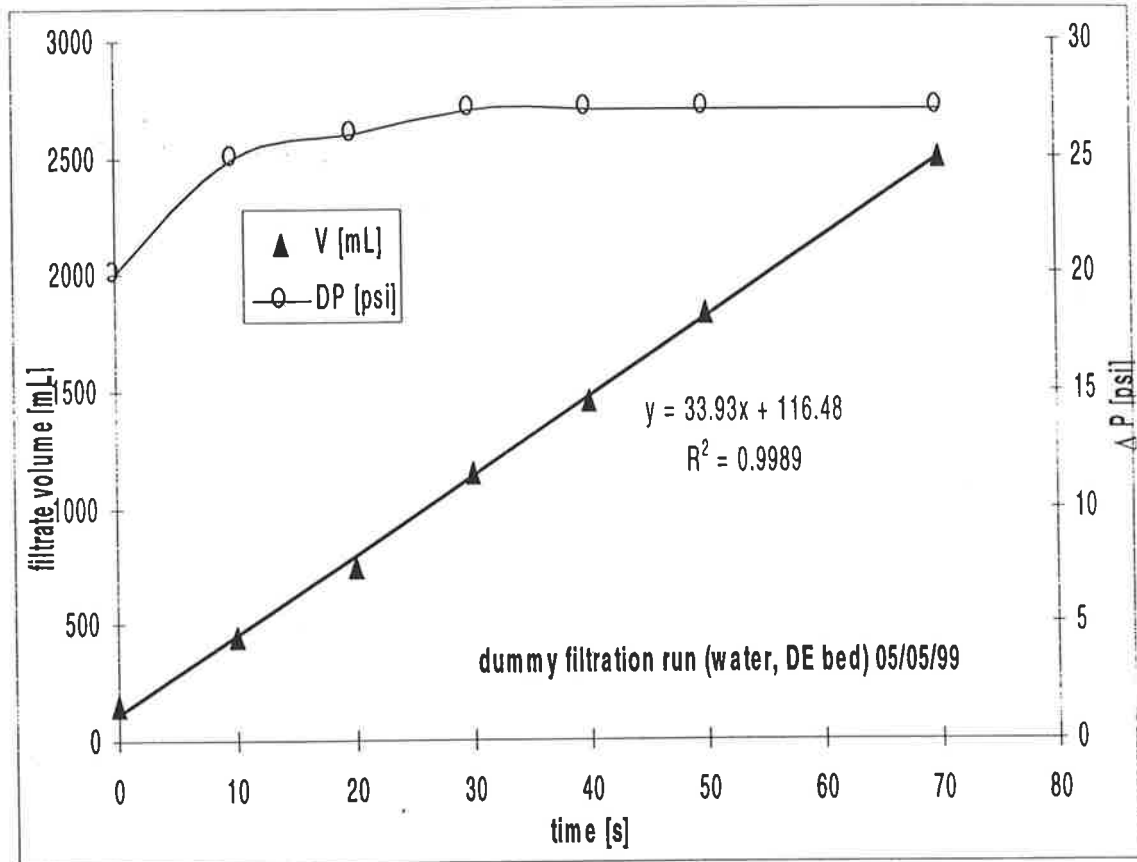


Figure 4.1 Preliminary trial run using tap water and (35 g) DE

($\epsilon_s = 0.861$ $K = 9.1 \times 10^{-13} \text{ m}^2$ $\alpha = 3.6 \times 10^9 \text{ m kg}^{-1}$).

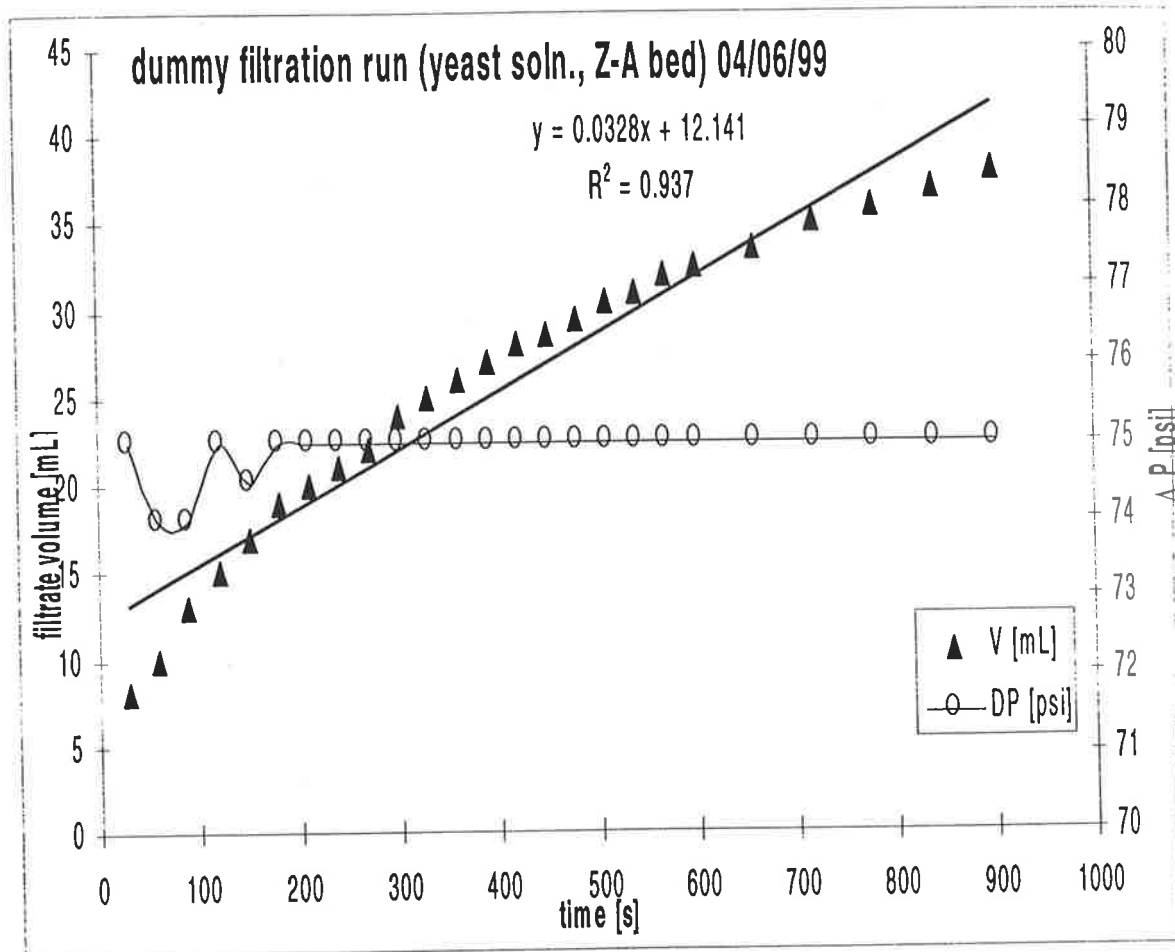


Figure 4.2 Trial run with yeast solution on a bed (35 g) of small grade zeolite-A
 $(\epsilon = 0.457 \quad K = 9.6 \times 10^{-17} \text{ m}^2 \quad \alpha = 9.1 \times 10^{12} \text{ m kg}^{-1})$

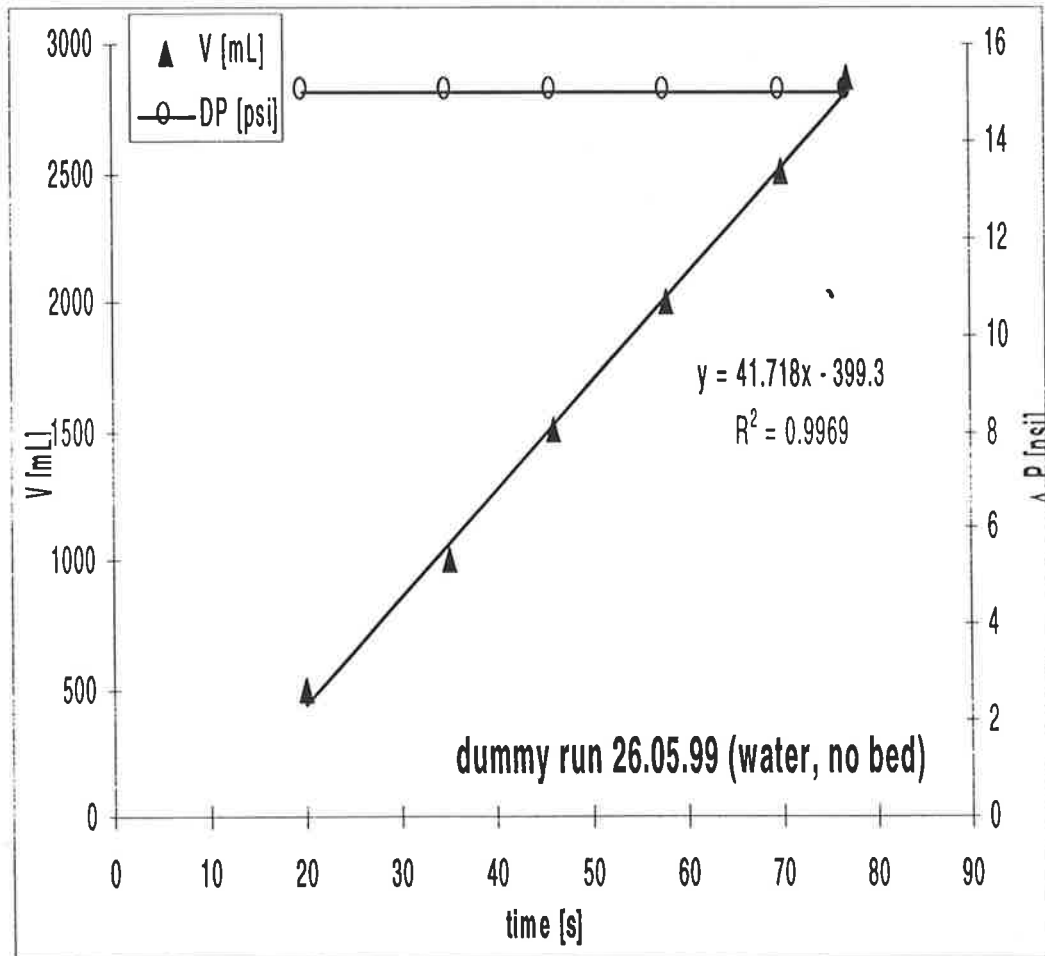


Figure 4.3 Trial run using tap water without filter medium (sintered plate only) to determine the bed resistance ($R = 11.0 \times 10^9 \text{ m}^{-1}$)

Table 4.1 Results of filtration of home-brewed *Black Rock Lager* beer using DE

Filter	Control	Filtrate
media weight (g)	na	35.14
filter pressure (psig)	na	30
filtration time (min.s)	na	02.00
filtration volume (L)	na	1.3
beaker mass (g)	33.24	9.61
sample volume (mL)	44.25	34.5
final mass (g)	76.81	43.94
sample mass (g)	43.57	34.33
sample density (kg m^{-3})	985	995
evap. beaker/solids (g)	34.9	10.8
solid content (g)	1.67	1.19
% solid content (wt %)	3.8	3.5
initial solid content (g)		1.67
initial solid content (wt %)		3.8
initial solid concentration (kg m^{-3})		37.6
final solid content (g)		1.19
final solid content (wt%)		3.5
final solid concentration (kg m^{-3})		34.5
solids removed (g)		0.47
% reduction of initial solids		8.2

Table 4.2 Results of filtration of home-brewed *Dark Ale* beer using DE

Filter	Control	Filtrate
media weight (g)	na	35.14
filter pressure (psig)	na	30
filtration time (min.s)	na	02.20
filtration volume (L)	na	1.0
beaker mass (g)	38.66	9.66
sample volume (mL)	31.50	33.5
final mass (g)	124.57	47.75
sample mass (g)	35.91	38.09
sample density (kg m^{-3})	1140	1137
evap. beaker/solids (g)	1.11	10.73
solid content (g)	3.1	1.07
% solid content (wt %)		2.8
initial solid content (g)		1.11
initial solid content (wt %)		3.1
initial solid concentration (kg m^{-3})		35.2
final solid content (g)		1.07
final solid content (wt%)		2.8
final solid concentration (kg m^{-3})		31.9
solids removed (g)		0.04
% reduction of initial solids		9.4

Table 4.3 Results of filtration of home-brewed *Dark Ale* beer using small grade zeolite-A

Filter	Control	Filtrate
media weight (g)	na	65.02
filter pressure (psig)	na	60 then 90
filtration time (min.s)	na	19.00
filtration volume (L)	na	0.5
beaker mass (g)	88.66	9.58
sample volume (mL)	31.50	36.75
final mass (g)	124.57	47.04
sample mass (g)	35.91	37.46
sample density (kg m^{-3})	1140	1019
evap. beaker/solids (g)	89.77	10.59
solid content (g)	1.11	1.01
% solid content (wt %)	3.1	2.7
initial solid content (g)		1.11
initial solid content (wt %)		3.1
initial solid concentration (kg m^{-3})		35.2
final solid content (g)		1.01
final solid content (wt%)		2.7
final solid concentration (kg m^{-3})		27.5
solids removed (g)		0.10
% reduction of initial solids		21.9

Table 4.4 Results of microbial analyses of home-brewed beers

Sample/Filter Medium	Mean* Viable Cell Count cell mL⁻¹	Standard Deviation cell mL⁻¹
Control: <i>Black Rock Lager</i>	4.82×10^6	2.29×10^5
<i>Black Rock Lager</i> beer filtered (35 g) DE	1.89×10^6	3.64×10^5
Control: <i>Dark Ale</i>	6.0×10^6	6.16×10^5
<i>Dark Ale</i> filtered with (35 g) DE	8.1×10^5	7.31×10^4
<i>Dark Ale</i> filtered (65 g) small grade zeolite-A	0	0

* Mean of three replicates

Table 4.5 Results of microbial analyses of filtrates of Yeast solution*

Media	Total Viable Yeast Cell Count** cell mL ⁻¹	Standard Deviation cell mL ⁻¹
cellulose (as cotton wool)	9.51 x 10 ⁶	6.77 x 10 ⁶
perlite	7.07 x 10 ⁶	5.26 x 10 ⁶
pumice	3.55 x 10 ⁶	2.16 x 10 ⁶
filter sand	7.43 x 10 ⁵	2.91 x 10 ⁴
precipitated silica	1.57 x 10 ⁵	6.12 x 10 ⁴
zeolite-A large grade (125-250 µm)	5.07 x 10 ⁴	2.65 x 10 ⁴
DE (as Celite 503)	0	0
zeolite-A small grade (63-125 µm)	0	0

* see Appendix C for details of solution preparation

** Mean of three replicates

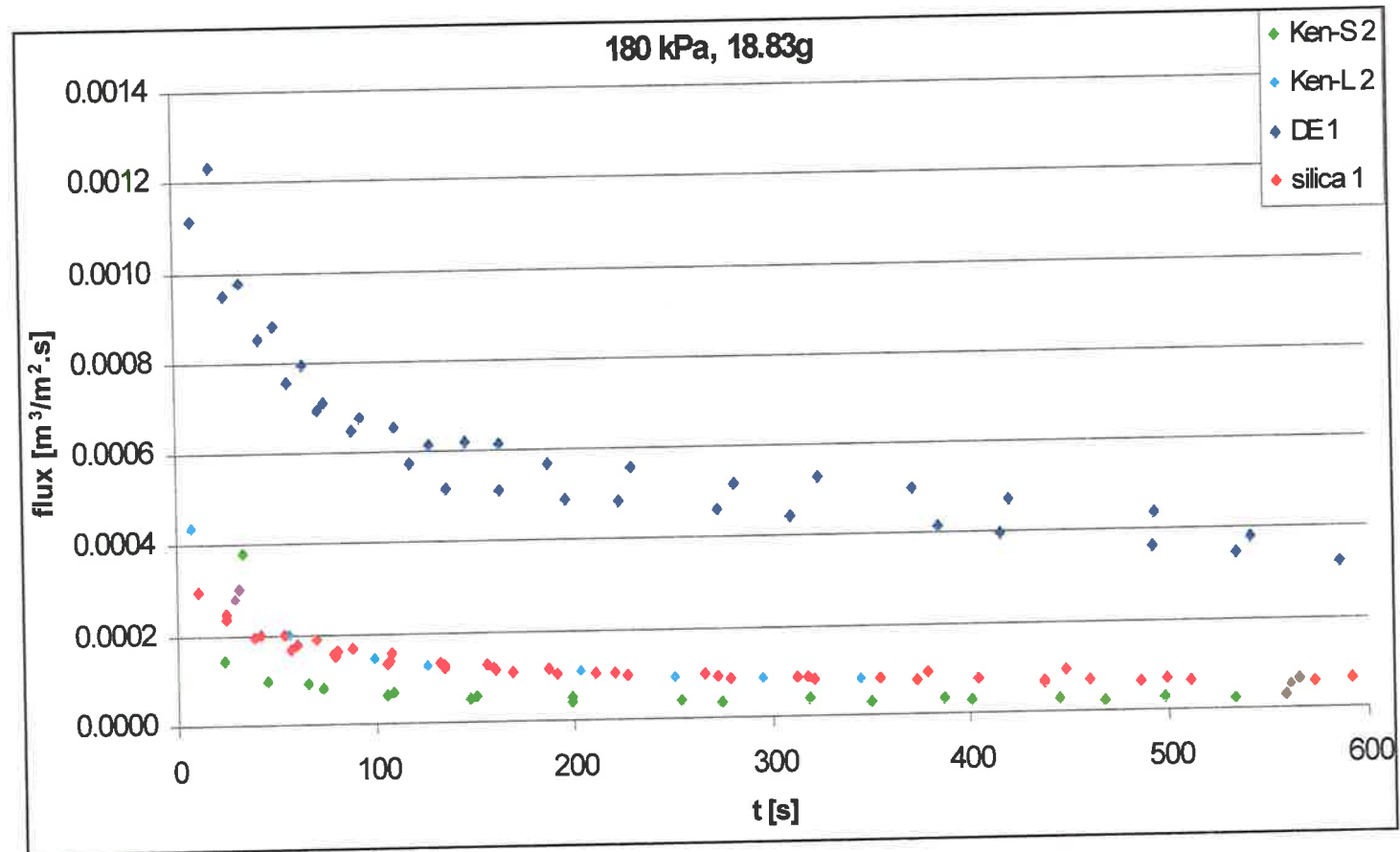


Figure 4.4 Results of flux-time experiments using 18.83 g media samples at 180 kPa

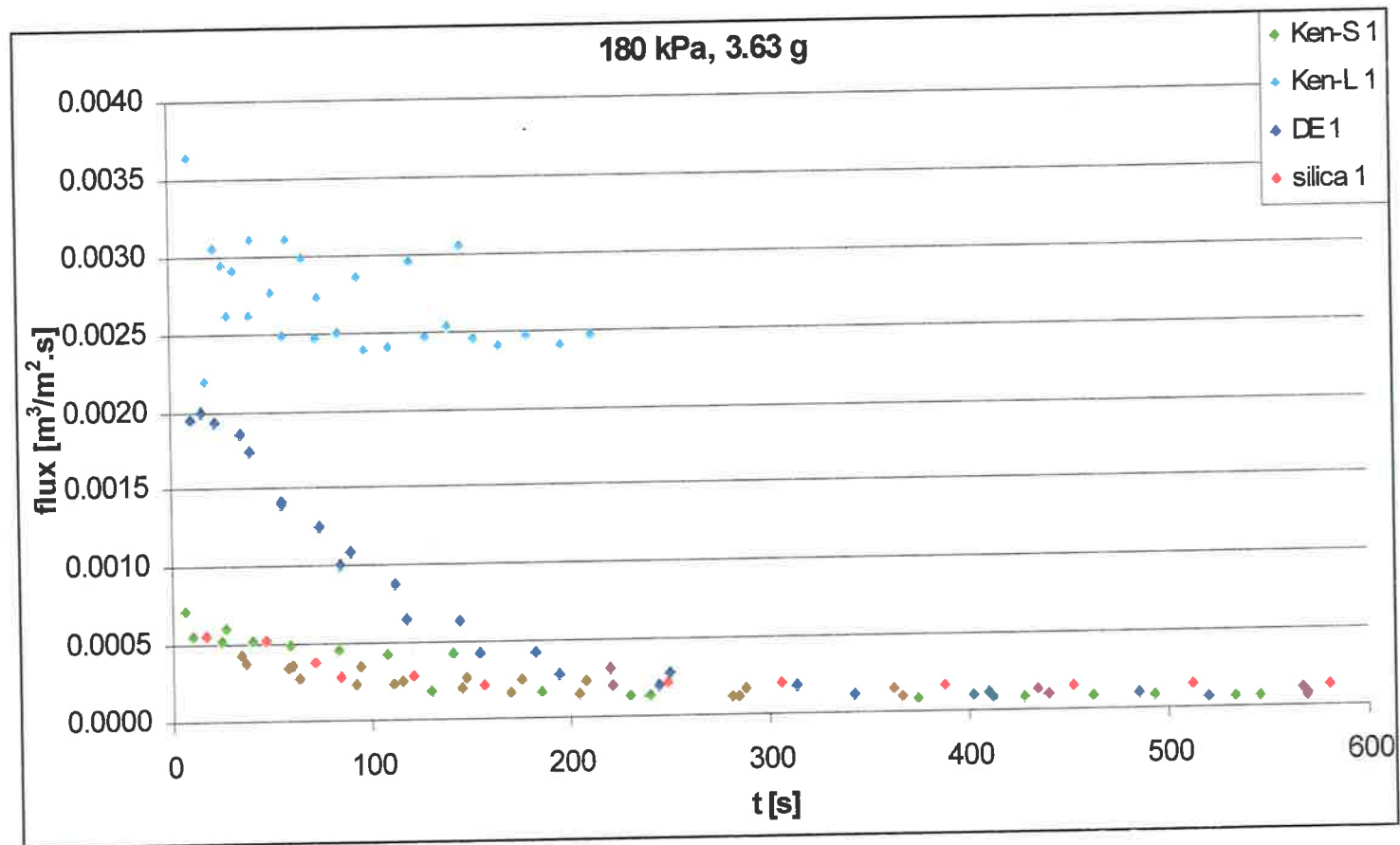


Figure 4.5 Results of flux-time experiments using 3.63 g media samples at 180 kPa

4.3 Analyses of Filtrates

4.3.1 European Brewing Convention (EBC) Units

Filtrates were kept at 2 to 4°C in the Lager cellar at Cooper's Brewer Ltd for a period of about 2 to 3 weeks until analyses in the commercial laboratory of the brewery. These filtrates were handled aseptically at all times.

Hazemeter readings on these filtrates showed that DE, small grade zeolite-A and the brewery Seitz filter gave acceptable haze levels of below 1 EBC (European Brewing Convention) unit. In most commercial beers, those filtrates with 0.8 EBC units or less are regarded as acceptable (Gan *et al* 1997; *pers. comm.* Dr Tim Cooper).

Figure 4.6 summarizes the average hazemeter reading (on three replicate filtrates) for each of the five media trialled and presents a comparison with the brewery's commercial Seitz filter (which itself uses DE). For pilot plant DE filtrates the mean haze reading is 0.6 EBC and those for the small grade zeolite-A of 0.8 EBC. Silica filtrates had a mean haze reading of just greater than 1 EBC, filter sand 5.2 EBC and the large grade zeolite-A a mean of 3.2 EBC. The commercial Seitz filtrates had a mean of 0.6 EBC. The pilot plant DE and small grade zeolite-A therefore gave very similar haze reducing capability as the commercial equipment of the brewery's Seitz filter.

Trials with the filter sand resulted in the formation of significant amounts of froth in the filtrates. The high and commercially unacceptable haze readings for large grade zeolite-A and silica probably indicate the presence of fine ($< 0.5 \mu\text{m}$) particles that have eluted from the bed.

4.3.2 Colour

The spectrophotometric analyses of filtrates is summarized as Figure 4.7. The mean absorbance reading on three replicates (produced from trials with a pressure gradient of 180 kPa and a filter bed mass of 18.83 g) for each filter medium is presented. The colour

of commercially produced beer filtrates (from the Seitz filter) gave an absorbance reading of 0.32.

Figure 4.7 highlights the fact that filtrates from the DE filter bed of the pilot plant had an almost identical mean absorbance reading (0.32) as the commercial “control” of the Seitz filter. Large grade zeolite-A and filter sand resulted in filtrates with an absorbance reading of 0.34 which compares favorably with DE and the commercial Seitz filter. The mean absorbance reading of filtrates from the small grade zeolite-A was 0.44, a value that is significantly greater than all other filtrates.

These spectrophotometric readings imply that a commercially unacceptable increase in colour is attached to filtrates using small grade zeolite-A. Interestingly, the colour of the filtrates from silica as the filter medium (with an absorbance reading of 0.29) was actually lighter than those of the Seitz filter.

4.3.3 pH

A summary of pH values of the resulting filtrate from each of the five filter media is presented as Figure 4.8. The figure shows that the pH value of the filtrate from small grade zeolite-A as filter medium increased from pH = 4 (i.e. the Seitz filter control value) to a pH value of 6. This represents an increase of two orders of magnitude in hydrogen ion concentration.

Generally, for commercial beers a narrow range of pH values from 3.5 to 4.5 pH units is desired (*pers. comm.* Tim Cooper). Clearly, the pH value of the filtrate from using the small grade zeolite-A as filter medium is significantly outside this range. Figure 4.8 shows that large grade zeolite-A also caused an increase in pH value (from about 3.9 to 4.3) of the filtrate but resulted in a value within the range suitable for commercial beer product. The pH value of the filtrate from both filter sand and silica is seen from Figure 4.8 to be equal to that from the Seitz filter control value of pH = 4 units.

Similar increases in pH value are reported with filtrates of white wine using identical small grade and large grade zeolite-A. Figure 4.9 (*adapted* from Figure 11 of Davey, Kadir and

Pecanek 1997) shows that small grade zeolite-A as filter medium for white wine resulted in a increase of pH value from 4 to 4.25 pH units, and; for the large grade zeolite-A from pH = 4 to pH = 4.6. For filter sand and DE the pH value of filtrates did not change for white wine – a similar result for that of the beer filtrates.

The resulting increase in pH value of the beer filtrates from small grade zeolite-A and large grade zeolite-A, and indeed of white wine filtrates (Davey, Kadir and Pecanek 1997), is accounted for by an increase in sodium ion concentration of the filtrate. The sodium ions are therefore leached from the zeolite-A media during filtration.

To investigate this phenomenon further trials were carried out with continuous filtration of beer to determine when sodium ion leaching from the filter medium would be exhausted. Results from these trials are presented and discussed in section 4.5 of this thesis.

4.4 Sensory Analyses of Filtrates

The Triangular Method of Analysis revealed brewery industry noses could differentiate between beer filtrates from DE and those from small grade zeolite-A as filter medium. The results from the Descriptive Comparison more clearly differentiated filtrates from the filter media.

4.4.1 Triangular Method

Filtrates of DE and small grade zeolite-A as filter media were analyzed using the Triangular Method (*see* Section 3.6). Results of the test are summarised in Table 4.6.

From the sixteen (16) assessors, ten (10) were able to distinguish the "odd" filtrate from the three given samples. This number of correct replies is greater than the minimum correct reply required to establish a significant difference between the two types of beer filtrates at 5% level of significance (*see* Table 4.7).

4.4.2 Descriptive Comparison

Filtrates of the five selected filter media and that from the Seitz filter were evaluated using the Descriptive Comparison (*see* Section 3.6) sensory test. The code for each of the filtrates as presented to the assessors and filtrate identity are listed in Table 4.8 and the mean rating for each of the characteristic attributes of the filtrates is presented as Table 4.9. Sixteen (16) noses evaluated the beer filtrates.

The mean rating values of this analysis is presented in a histogram as illustrated in Figure 4.10.

The DE-filtered beer is the highest rated filtrate for aroma with a mean rating of 5.60 followed in descending order by filtrates of filter sand, large grade zeolite-A, Seitz filter, silica and the least rated is the filtrate of small grade zeolite-A with mean rating of 4.13.

The best colour rating was that of the filtrate of filter sand with mean rating of colour = 6.80. This is followed by filtrates of DE, then equal mean ratings for Seitz filter and large grade zeolite-A filtrates (colour = 6.40), followed by silica and lastly the small grade zeolite-A with a mean rating of colour = 4.73. These results are supported by the spectrophotometric analyses of the filtrates, where the absorbance of the filtrate of silica is lower than that of the other filtrates except that of the filtrate of small grade zeolite-A which is higher by about 0.12 from the other absorbance readings.

Filtrate of large grade zeolite-A has the highest mean rating for clarity of 7.60. This is followed in descending order by filtrates of DE, silica, equally rated Seitz filter and filter sand and lastly small grade zeolite-A with mean rating of 6.67. These results are not in agreement with hazemeter readings obtained in the laboratory. The filtrates of filter sand and large grade zeolite-A have high haze level contents and the small grade zeolite-A with EBC units within the acceptable level but visual analysis of these filtrates gave different results as evident from the sensory ratings.

The highest rated filtrate for its taste is that of the filter sand (mean rating of 5.80) followed in descending order by large grade zeolite-A, DE, Seitz filter, silica and lastly with the filtrate of small grade zeolite-A with mean rating of 3.89.

Among the filtrates analysed, the most preferred for its drinkability and overall impression is the one filtered with filter sand (442) with mean rating of 5.73 and the least preferred are the small grade zeolite-A (146) and silica (146) with equal mean ratings of 4.00. The other filtrates rated in ascending order as Seitz filter (control), large grade zeolite-A and DE.

From the ranking of each characteristic according to the average intensity of the perception of the assessors (Table 4.9), it can be seen that the filtrate of filter sand has the best attribute overall, with high attribute ratings except for its clarity. Large grade zeolite-A is comparable to the existing medium (DE) with the alternative medium being more preferred in clarity and taste.

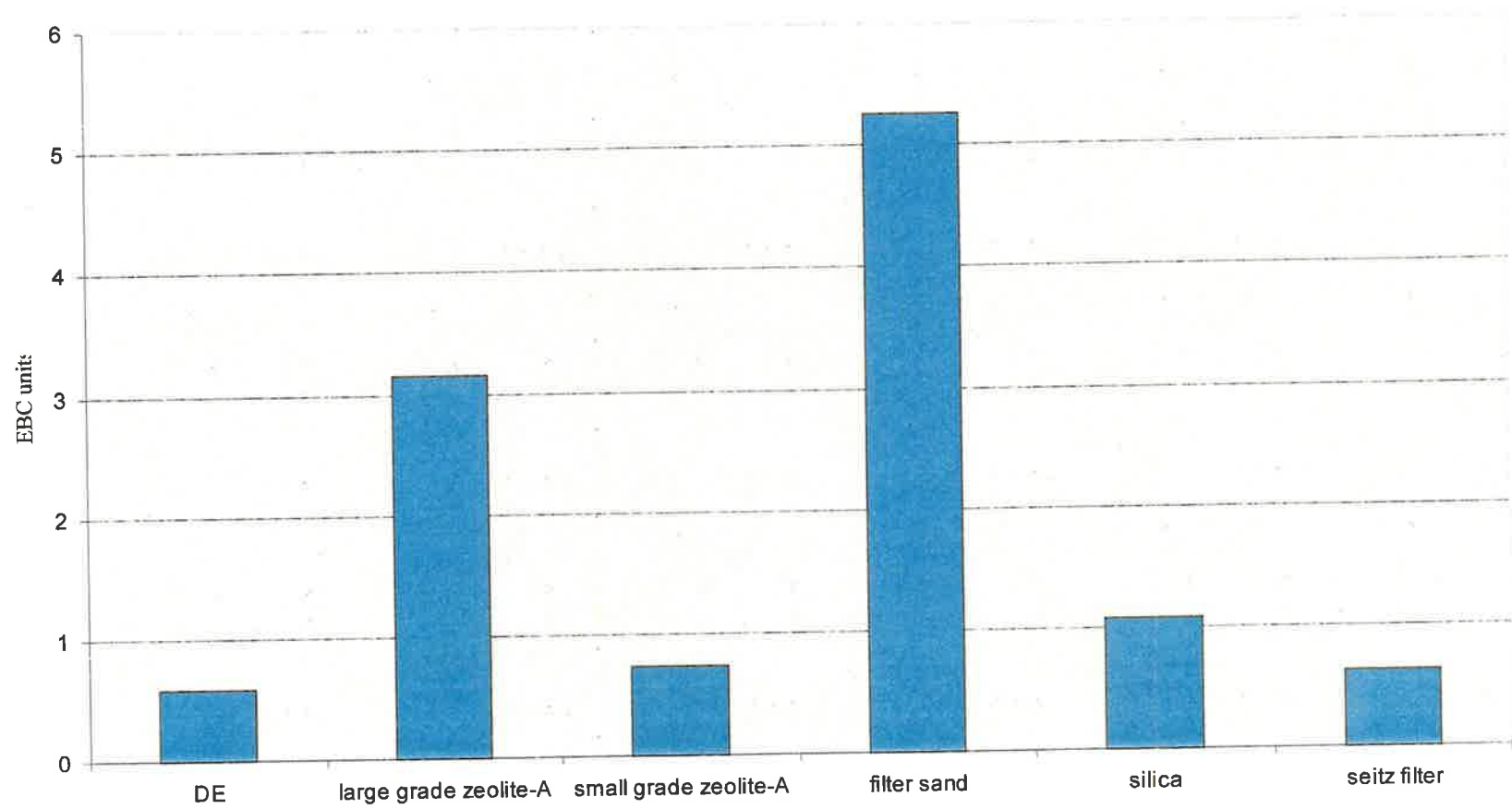


Figure 4.6 Hazemeter reading expressed as EBC units (average of 3 readings)

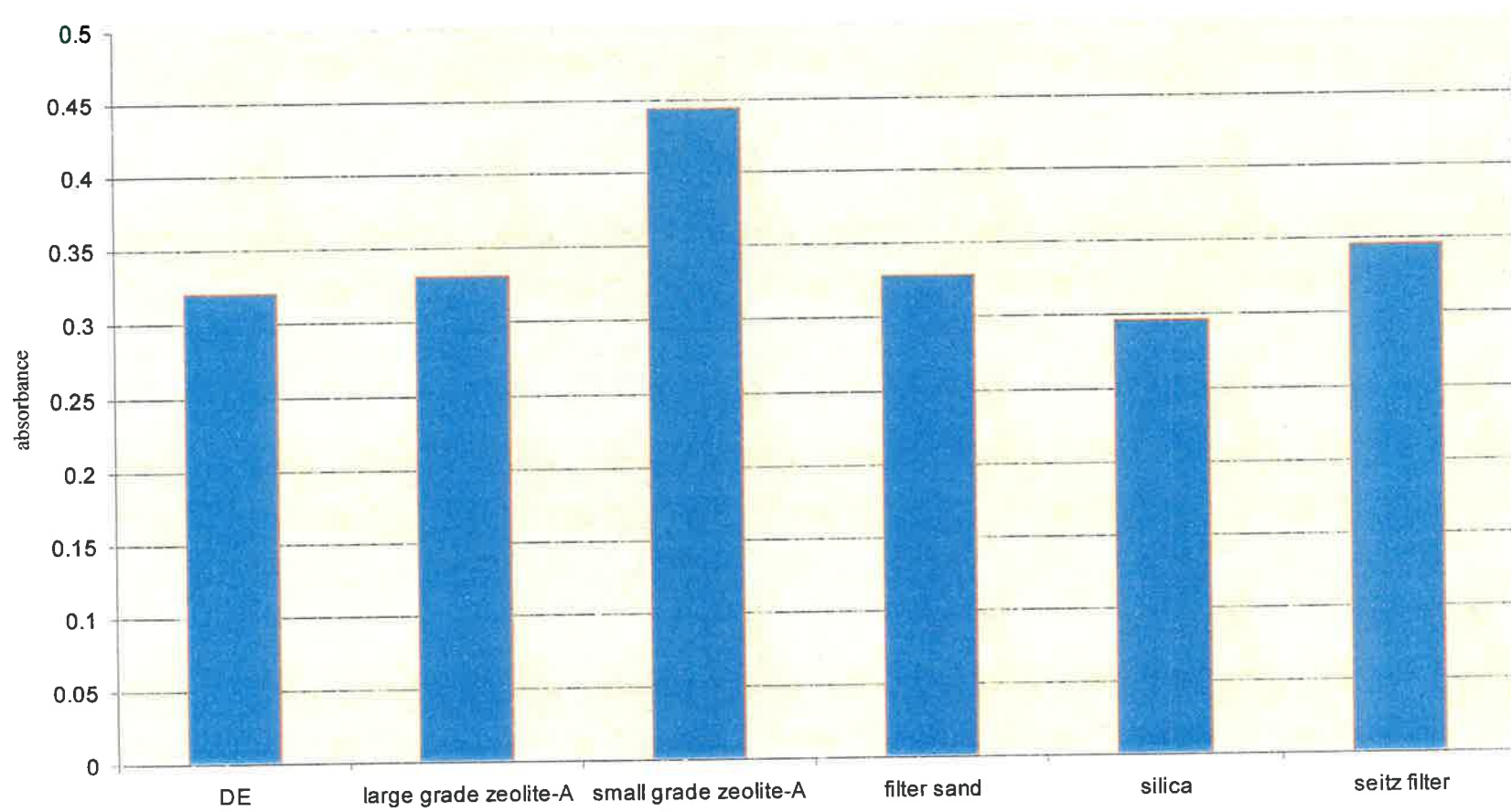


Figure 4.7 Spectrophotometric Analysis (mean of 3 readings)

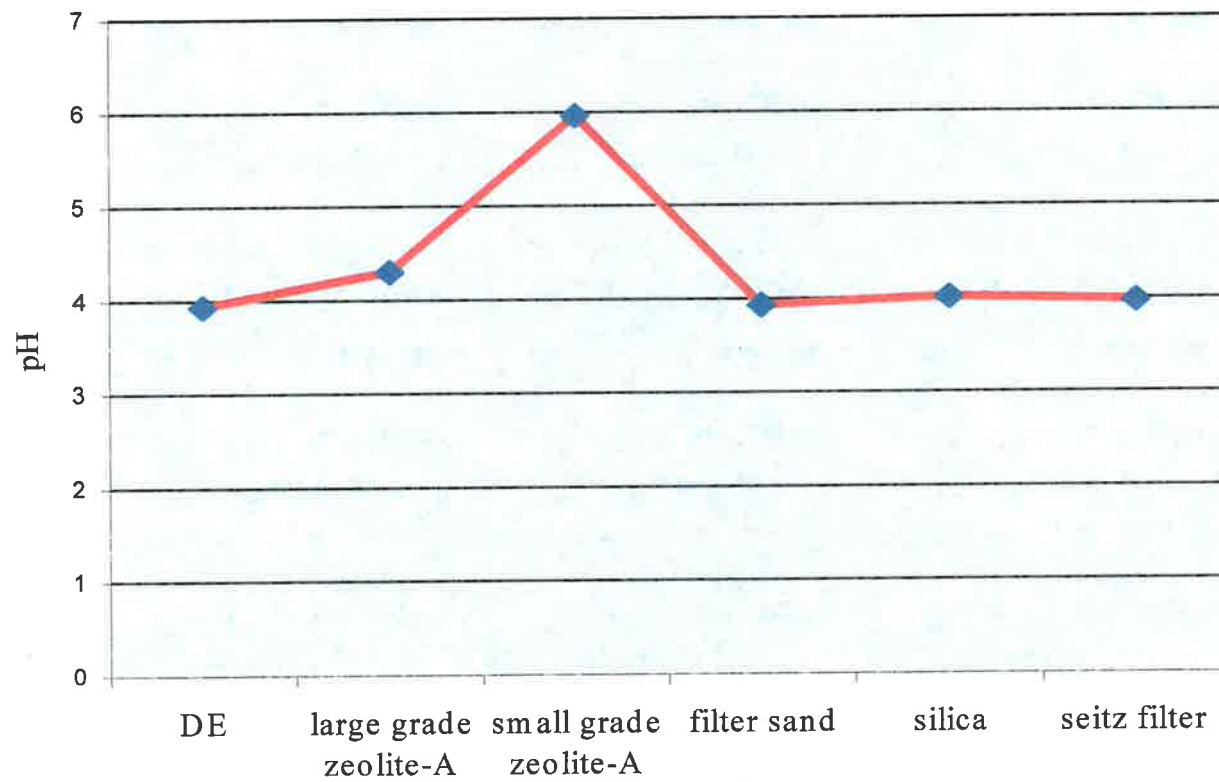


Figure 4.8 Summary results of pH of filtrates measured at 20°C

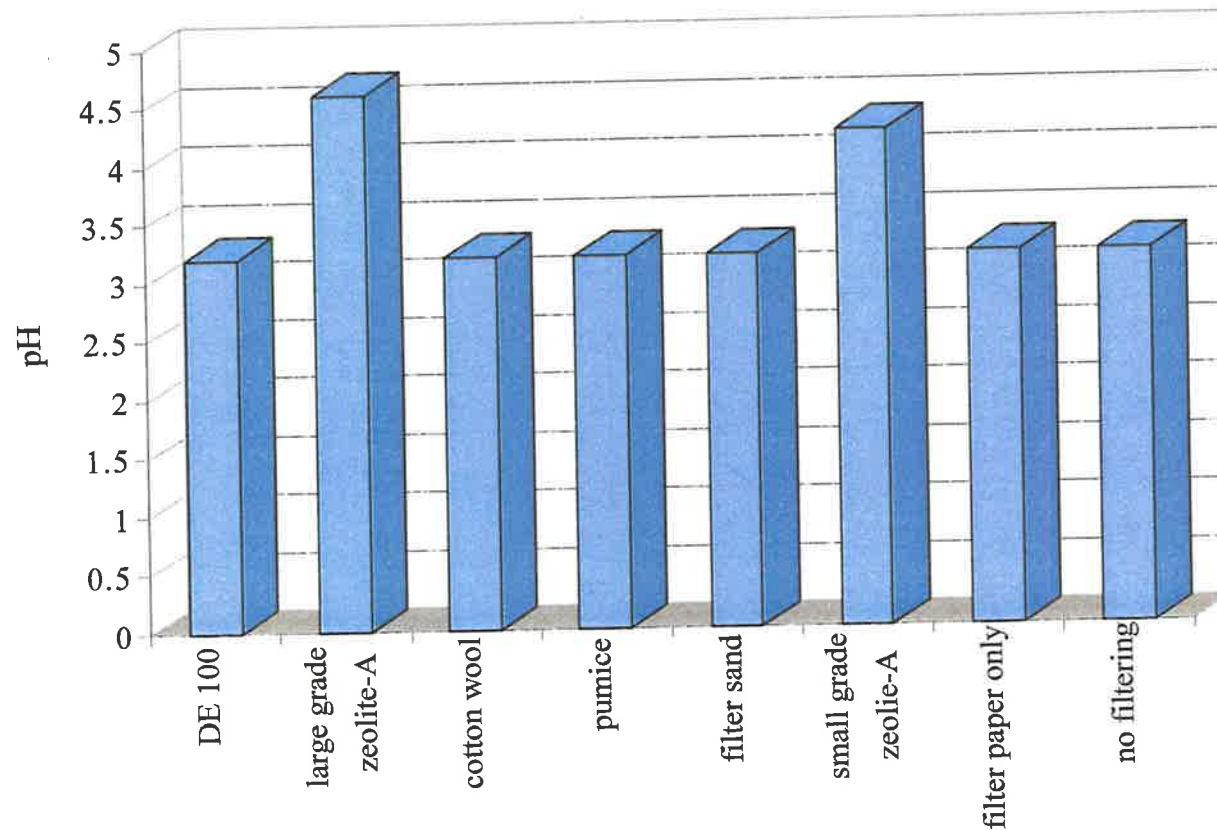


Figure 4.9 Summary result of the pH of wine filtrates
(adapted from Davey, Kadir and Pecanek 1997)

Table 4.6 Summary of results of the Triangle Method

Correct Reply	Wrong Reply
C/A*	C/G
D/B	F/S
D/L	K/P
J/D	M/B
Mc/D	P/M
S/A	S/N
M/J	
P/J	
R/D	
V/P	

* identifier for brewery assessor (nose)

Table 4.7 Probability levels for Triangular Method (one sided, $p = 1/3$) as used in the brewing industry*

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TABLE I Minimum Numbers of Correct Replies to Establish Significance at Various Probability Levels for the Triangular Test (One-Sided, $p = 1/3$) ^a											
Number of Replies	Minimum Number of Correct Replies for a Significance Level of $\alpha \leq$			Number of Replies	Minimum Number of Correct Replies for a Significance Level of $\alpha \leq$			Number of Replies	Minimum Number of Correct Replies for a Significance Level of $\alpha \leq$		
	0.05	0.01	0.001		0.05	0.01	0.001		0.05	0.01	0.001
5	4	5	...	37	18	20	22	69	31	33	36
6	5	6	...	38	19	21	23	70	31	34	37
7	5	6	7	39	19	21	23	71	31	34	37
8	6	7	8	40	19	21	24	72	32	34	38
9	6	7	8	41	20	22	24	73	32	35	38
10	7	8	9	42	20	22	25	74	32	35	39
11	7	8	10	43	20	23	25	75	33	36	39
12	8	9	10	44	21	23	26	76	33	36	39
13	8	9	11	45	21	24	26	77	34	36	40
14	9	10	11	46	22	24	27	78	34	37	40
15	9	10	12	47	22	24	27	79	34	37	41
16	9	11	12	48	22	25	27	80	35	38	41
17	10	11	13	49	23	25	28	81	35	38	41
18	10	12	13	50	23	26	28	82	35	38	42
19	11	12	14	51	24	26	29	83	36	39	42
20	11	13	14	52	24	26	29	84	36	39	43
21	12	13	15	53	24	27	30	85	37	40	43
22	12	14	15	54	25	27	30	86	37	40	44
23	12	14	16	55	25	28	30	87	37	40	44
24	13	15	16	56	26	28	31	88	38	41	44
25	13	15	17	57	26	28	31	89	38	41	45
26	14	15	17	58	26	29	32	90	38	42	45
27	14	16	18	59	27	29	32	91	39	42	46
28	15	16	18	60	27	30	33	92	39	42	46
29	15	17	19	61	27	30	33	93	40	43	46
30	15	17	19	62	28	30	33	94	40	43	47
31	16	18	20	63	28	31	34	95	40	44	47
32	16	18	20	64	29	31	34	96	41	44	48
33	17	18	21	65	29	32	35	97	41	44	48
34	17	19	21	66	29	32	35	98	41	45	48
35	17	19	22	67	30	33	36	99	42	45	49
36	18	20	22	68	30	33	36	100	42	46	49

^aThe values in this table were calculated from the exact formula: binomial law for parameter $p = 1/3$ with n repetitions (replies). When the number of replies is larger than 100, numbers of required correct replies may be obtained from the following formula based on the approximation of the binomial law by the normal law, with a maximum error equal to one unit: $X = 0.4714 \times \sqrt{n} + [(2n + 3)/6]$ where $x = 1.64$ for $\alpha \leq 0.05$; 2.33 for $\alpha \leq 0.01$; and 3.10 for $\alpha \leq 0.001$ (Ref. 5). The minimum number of correct replies is X if X is a whole number, or the next higher integer if X is not a whole number. Tables for significance levels other than those listed here may be found in Refs. 2 and 5.

* supplied by Cooper's Brewery Ltd.

Table 4.8 Codes and description used for the Descriptive Comparison

Sample Number	Description
146	silica
552	DE
442	filter sand
579	small <i>Ken</i> *
857	Seitz filtered
361	large <i>Ken</i>

* To disguise zeolite-A as a filter medium and to preserve the confidential nature of this study, zeolite-A was referred to as *Ken* in all trials that were carried out away from the laboratories of the University.

Table 4.9 Mean rating for Descriptive Comparison

Simple Descriptive Comparison for the Evaluation of Beer Filtrates					
Mean Rating*					
Sample Code	Aroma	Colour	Clarity	Taste	Drinkability/ Overall impression
146	4.33	6.29	7.29	4.00	4.00
552	5.60	6.47	7.47	5.29	5.69
442	4.75	6.80	7.07	5.80	5.73
579	4.13	4.73	6.67	3.89	4.00
857	4.56	6.40	7.07	4.89	5.27
361	4.60	6.40	7.60	5.33	5.50

*mean rating on 16 brewery assessors (noses)

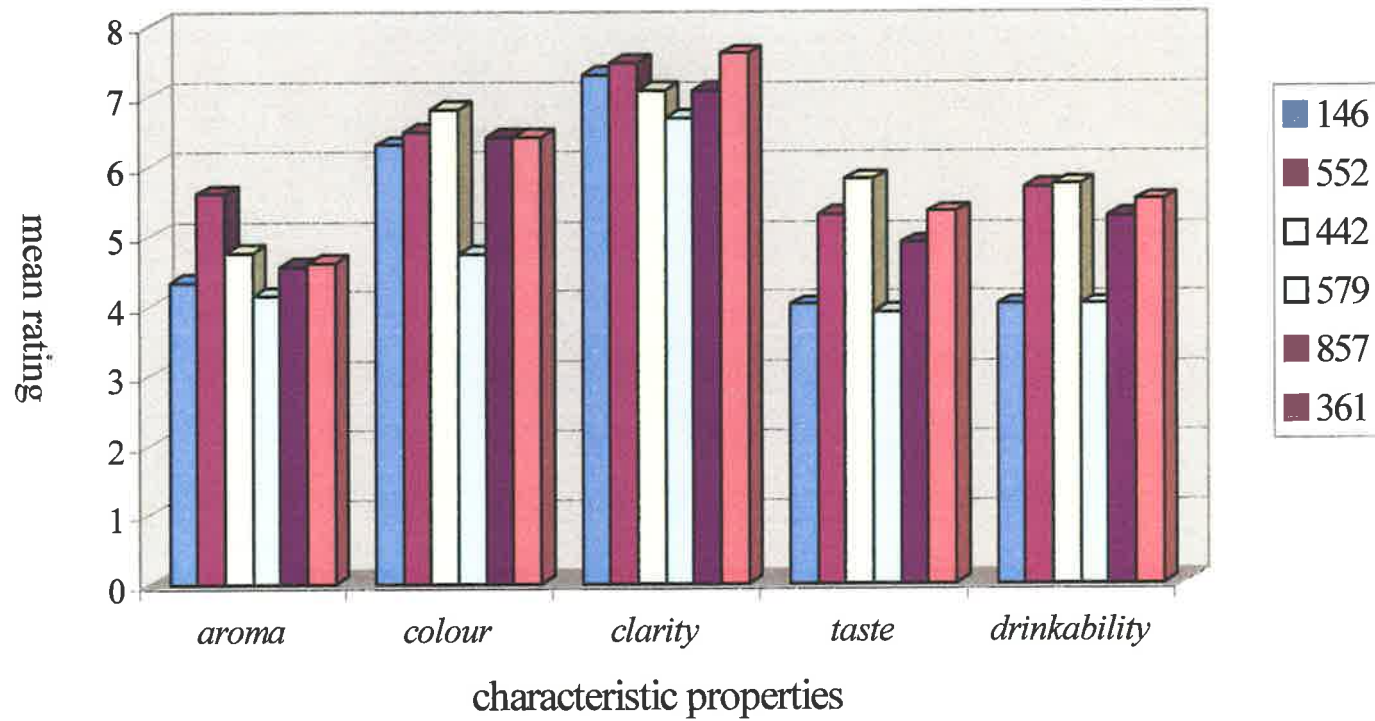


Figure 4.10 Summary histogram of the Descriptive Comparison of filtrates

4.5 Ion-Exchange

Further experimental study was conducted to determine the factors that might be causal in the increased pH of the beer filtrates. The assumption was made that:

- alkaline (sodium) species leaching out of the zeolite structure gave rise to the increase in pH.

The effect of sodium on the pH of the filtrate was therefore investigated. During beer fermentation, the pH of the beer is reduced as a result of the increased production of the positively charged non-microbiological particles (NMPs) (Leather, Dale and Morson 1997).

With filtration however, these positively charged NMPs are removed from the beer by substitution. The cations located in the pores of the zeolite filter medium, in this case sodium cations (*see* Section 2.4 pp. 11 and 16), are substituted with the NMPs and the sodium cations are washed away to become part of the filtrate.

To validate the assumption that sodium cations are washed through the filter and into the filtrate from the zeolite-A filter bed an experimental design was considered and implemented. At recorded time intervals, corresponding to predetermined volumes of filtrate, the pH of the beer filtrate was monitored. The aim was to test whether the sodium ion release could be exhausted from the filter media. Filter beds (18.83 g) of both the small and large grade zeolite-A with a pressure driving force of 180 kPa were experimentally investigated. Four (4) replicates were used and the pH of the filtrate monitored for between 8 and 16 h of continuous filtration to determine if all sodium could be exhausted from the medium.

A practical constraint to further study soon emerged because the filter cake became progressively clogged with yeast and other solid particles from the beer haze before a detectable change in pH of the filtrate could be detected.

One reason why larger numbers of sodium ions leach from an equivalent mass of 18.83 g of the small grade (63 to 125 μm particle diameter) zeolite-A filter cake (resulting in a pH increase of 2 pH units of the filtrate), in comparison with the smaller number of sodium ions that leach from the large grade (125 to 250 μm particle diameter) zeolite-A filter cake (resulting in an increase of less than 0.5 pH units) could be related to a difference in residence time of the beer in the two filter cakes.

The length of the filter path for passage of beer in the small grade zeolite-A would presumably be significantly greater than with the large grade material. There is therefore an overall larger surface area of filter medium in contact with the beer with the small grade material together with a greater residence time of the beer compared with the large grade material. The release of sodium ions therefore appears to have both a mechanical and a chemical basis in giving rise to increasing the pH of commercial beer filtrates.

One approach to this as yet unresolved problem might be to prevent the sodium cations leaving the filter medium with the use of a carefully selected chelating agent. This agent might be added during preparation of the wetted slurry of the medium. The desired outcome is that the sodium will be trapped to the chelating agent whilst the positively charged particles of the beer haze are attached within the zeolite-A framework. It should be reiterated that the increase in pH of the beer filtrates from the large grade zeolite-A filter cake was less than 0.5 pH units. A judiciously selected particle size for zeolite-A filter medium might be possible to limit this in the first instance.

4.6 Concluding Remarks

Experimental data have been obtained for the filtration characteristics of several media, and resulting filtrates, using the procedures and pilot plant described in Chapter 3.

A comparison of results from a commercial mix of DE with selected zeolite-A as filter media for the removal of haze from beer has shown that large grade zeolite-A (particle size 125-250 μm) could provide a practical alternative to DE in the brewing industry. The source of the presence of sodium ions - resulting in a detectable increase in pH - of the filtrate is attributed to the zeolite-A media. The particular zeolite-A used was selected as it

was judged as nearly the same as that manufactured within the Department of Chemical Engineering, Adelaide University from naturally occurring deposits of kaolin.

It is not known whether a range of alternative zeolites might also provide a practical alternative to DE. Present understanding must therefore be said to be limited. Alternative zeolites for the removal of haze from beer would need to be evaluated experimentally. The pilot plant and procedures developed for this study would be readily applicable for such an undertaking.

A deciding factor in the choice and use of zeolites as filter media for the removal of haze in beer (and the polishing of wine) might ultimately be that the DE currently used is classified as a serious carcinogen with its attendant health risks. A trade-off therefore of performance of alternative filter media with perceived health risks is most probable.

There is no evidence available to show that zeolites have been studied as an alternative to DE for the removal of haze. Therefore the findings reported in the present study, together with earlier findings from the polishing of white wine, strongly indicate the basis for development of IP.

CHAPTER 5 - CONCLUSIONS

The study of zeolite-A as a substitute filter medium to diatomaceous earth (DE) in the filtration of beer haze has given rise to the following conclusions:

1. The search for an alternative filter to DE in both the brewing and wine industries is both timely and increasingly necessary given the classification of commercial mixes of DE as "Human Carcinogen Category 1". There is no evidence to show that zeolites have been studied as an alternative filter medium to DE for the removal of haze in beer.
2. A pilot plant can be reliably used to practically simulate brewery industry process operations of: filter type, bed (cake) depth, preparation protocols and pressure driving force.
3. Zeolite-A of a type produced in the laboratories of Adelaide University from conversion of kaolin is available commercially. It is similar to brewery DE in terms of bulk physical characteristics. Importantly it can be readily substituted for DE without the need for significant changes in brewery industry process equipment and protocols for preparation. Zeolite-A has a lower packed-bed voidage than commercial DE (respectively, 0.457 and 0.861 with DE as Celite 503). Whilst DE is inert, the particular zeolite-A appears to exhibit adsorptive and ion exchange properties.
4. The adsorptive and ion exchange properties of filter beds of zeolite-A in the pilot plant give rise to an increase in pH of the filtrate. Although small grade zeolite-A (63-125 μm mean particle diameter) effectively removed haze from beer to a commercially desired clarity, it adversely resulted in an increase of about 2 pH units of the filtrate. Large grade zeolite-A (125-250 μm mean particle diameter) also resulted in an increase in pH of the filtrate (of about 0.5 pH units) but gave similar fluxes as obtained with DE (290 and 390 $\text{mL m}^{-2} \text{s}^{-1}$ respectively) at equivalent brewery process conditions (35 g filter media and 206 kPa). DE, filter sand (200 μm mean particle diameter) and silica did not affect a measurable change in pH of the filtrates.

5. The release of sodium ions appears to have both a chemical and a mechanical basis in giving rise to an increase in the pH of commercial beer filtrates.
6. Industry indices for sensory analyses highlighted significant and adverse differences in consumer preference for beers filtered using small grade zeolite-A over DE. Small grade zeolite-A therefore is not suitable as a substitute for DE in the removal of haze in beer.
7. Visual and sensory analyses of filtrates from large grade zeolite-A filter beds resulted in the highest rating on industry indices by industry noses. Filtrates from large grade zeolite-A compared well to filtrates from DE and the brewery (control) Seitz filter. Therefore expectations of consumers are likely to be met with beer filtered using large grade zeolite-A.

RECOMMENDATIONS FOR FURTHER STUDY

1. Large grade zeolite-A should be further studied as a practical alternative to diatomaceous earth (DE) for the removal of haze in beer. Importantly zeolite-A can be readily substituted for DE without the need for significant changes in brewery process equipment and protocols. An understanding of the mechanical and chemical factors that give rise to the adsorptive and ion exchange properties should be emphasised. However, a trade-off of performance of zeolite-A as an alternative filter media with the health risks associated with the dry form of DE is most probable.

Because it is not known whether a range of alternative zeolites might also provide a practical alternative to DE present understanding must therefore be said to be limited. Alternative zeolites for the removal of haze from beer would need to be evaluated experimentally. The pilot plant and procedures developed for this study would be readily applicable.

2. There is no evidence available to show that zeolites have been studied as an alternative to DE for the removal of haze in beer (or wine). Therefore the findings reported in the present study, together with earlier findings from the polishing of white wine, strongly suggest development of IP patent(s) be undertaken.

APPENDIX A: A definition of some important terms used in this study

body feed	filter aid that is added to the beer to be filtered which then builds up the filter cake. As the filtrate passes through the filter, the body feed deposits on the filter surface keeping pores open.
bright	a sparkling beer that is very clear (very small haze). Bright beer is age stable and chill stable.
cake (bed)	solids stopped at the surface of the filter medium that pile upon one another to form a cake (bed) of increasing thickness.
cake resistance	resistance to flow through a filter media bed.
clarification	process of removing fine suspended substances from rough beer.
centrifuge	a machine designed to separate excess yeast from beer.
diatomite	a naturally occurring mineral derived from fossilised marine diatoms.
drinkability	a professional index for overall impression of a beer product.
EBC	<i>European Brewing Convention</i> . A unit used for the haze content of beer. The lower the value the better the clarity. A typical value for filtered industry beers is < 1 EBC.
egg pump	vapour pressure above the surface of a liquid in a leak-proof vessel is used to force the liquid through related pipe work.
fermentation	the action of yeast converting sugars to alcohol.

filtrate	liquid that has been filtered in a filter media.
filtration	a term for the unit operation in the process of removing solid particles from beer using a filter unit and septum.
finings	added substances which encourage flocculation of colloidal particles (example bentonite, gelatine and egg white).
flux	quantity of filtrate per unit area of filter media per unit time.
haze	cloudiness in beer, common causes are starches, protein and yeast.
IARC	<i>International Agency for Research on Cancer.</i>
IDPA	<i>International Diatomite Producers Association.</i>
kaolin	a naturally occurring form of clay.
kieselguhr	an alternative term for diatomaceous earth (DE).
lager cellar	where the beer is stored at low temperature before filtration.
nose(s)	an experienced and professional expert used to assess sensory characteristics of beers (also wines).
polishing	a form of clarifying that covers the final clarification step.
pre-coat	a thin layer of filter aid added to the filter support before filtration.
protein	amino acid sequence connected by peptide bond; causes haze.
septum (filter)	membrane that supports the filter bed/cake. Usually a finely perforated screen or a sintered plate.

<i>SOP's</i>	<i>Safe Operating Procedures</i> – a hazard and safe operating checklist and resulting protocol for start up, operation, shutdown and maintenance.
sterile	absence of all viable micro-organisms.
wort	clear grain digestate, the clarified extract solution of malt.
yeast	<i>Saccharomyces cerevisiae</i> - responsible for conversion of wort to beer.
zeolite	microporous, crystalline material composed primarily of SiO_4 and AlO_4 . Zeolites have a three-dimensional, crystalline framework of tetrahedral silica aluminium anions strongly bonded at all corners.

APPENDIX B: SOP's for the pilot plant

PART 1: Safe Start-Up Procedure for the Pilot Plant

(Describes concisely and in a step by step manner exactly how to safely start the equipment, apparatus or process).

1. Ensure that all the valves are closed, the filter unit disassembled and pressure vessel open.
2. Fill the vessel with the process liquid. Place gasket and lid aligning it with the notch. Make sure the bolts are double-washed and tightened opposite sides first. Alternatively, vessel can be filled with the process liquid via V6 using V4 to aid the flow. Close V4 and V6 when done.
3. Prepare the slurry by measuring the amount of filter media and mixing it with reverse osmosis water.
4. Wrap the sintered metal plate with wet Whatman #1 filter paper. Place it in the inside bottom of the filter unit, then fit in the O-ring.
5. Hold the polycarbonate cylinder on the O-ring, push down firmly as the slurry is poured in. Continue holding the cylinder to avoid leakage of the slurry.
6. Place the top section of the filter unit on cylinder top. Bolt and Tighten. Release hold on cylinder.
7. Secure blast shield. Ensure that all N₂ lines and beer lines are tightly attached and secure.
8. Check all valves. Set V2 to process line and ensure that V3, V4, V6 and V7 are shut.
9. Adjust V1 to be about 20% above the required ΔP (as measured on cylinder gauge). DO NOT EXCEED 1000 kPa. STAY BEHIND SHIELD FROM THIS POINT, UNTIL SYSTEM IS PURGED AND V1 IS OFF AGAIN.
10. Adjust V3 until pressure vessel gauge displays required ΔP .
11. Check for leaks. If leaks are present, shut down, purge and tighten bolts then recommence start up.
12. Ensure filter outlet is correctly placed (drain/sample container)
13. Carefully open V7.

PART 2: Safe Shut Down Procedure

(Describes concisely and in a step by step manner exactly how to safely shut the equipment, apparatus or process down).

Do Behind Shield

1. Emergency Shutdown: close V7
Normal Shutdown: close V3
2. Close V1 to shut off N2 supply.
3. Carefully switch V2 to blowdown line.
4. Carefully open pressure vessel blowdown valve (V4).
5. Check that V2 is on blowdown and V4 is open. System is now depressurised.
6. Undo beer lines.
7. Unbolt filter unit and pressure vessel lid. Empty and clean vessel.
8. Undo filter unit exit line. With a pencil or glass stirring rod, carefully push up on the sinter plate through the outlet fitting. Polycarbonate cylinder will rise with it.
9. Take cylinder and plate/bed to the bench. Detach plate/bed from the cylinder. If bed is not of interest, dispose of now- bed is relatively harmless when wet.
10. Clean filter unit.

PART 3: Maintenance Procedure of the Pilot Plant

(Describes concisely and in a step by step manner exactly how to safely maintain the equipment, apparatus or process).

Daily Maintenance

Clean with hot water and rinse with RO water.

Soak sinter plate in very hot water. Rinse thoroughly.

Check for integrity of rubber components (O-rings, gasket) and plastic tubing.

Weekly Maintenance

Check metal components for signs of corrosion. Replace corroded parts if necessary.

Rinse all equipment with sodium metabisulphite solution.

Rinse at least twice with water.

Yearly or Maintenance as Required

Replace O-rings and gasket.

Check gauges and valves for internal damage/corrosion replace if necessary.

PART 4: Requirements

(Describes concisely and exactly what requirements, equipment or materials, including personal protection are required for this apparatus or process).

Safety glasses

Latex gloves when handling filter media (wet or dry)

Dust masks when handling/exposed to dry filter media

Airtight containers for storing media

Allen keys

Small shifter spanners

Screwdriver (medium, standard)

Medium shifter

(When handling dry filter media, minimise the time whenever possible. Media are almost entirely safe when wet/slurried thoroughly).

PART 5: Safety and points to note

(Describes concisely and exactly what hazards are present during any phase of operation of this apparatus or process. Includes personal protection that may be required).

Filter Media

Particles of very fine size, such as DE used are classified as carcinogenic by WHO.

Wear dust masks when handling dry.

Wear safety glasses at all times.

Store in airtight containers.

Handle wet whenever possible.

Avoid contact with the skin.

Wash hands thoroughly after use.

Dispose of wet media as soon as possible.

Possible exothermic reaction with skin moisture if handling with bare hands.

Pressure

Follow SOP to avoid spraying or loss of vessel contents or blowout.

Follow maintenance procedure regularly.

Off-Site Work

Be aware of the fire escape, medical equipment and exits at all times.

Check in and check out every day.

Follow site emergency procedure if necessary.

Noise

Use earplugs if discomfort occurs.

PART 6: Future Developments

(Describes what future developments or improvements are suggested which could make the apparatus safer or better).

Fix the persistent small-scale leak at the pressure relief valve (V5).

Provisions for airtight containers.

Insulation of lines and filter (possible filter if possible)

More spare bolts and wingnuts for different filter cylinder sizes.

PART 7: Maintenance Record

(Record of the maintenance carried out or modifications made to the apparatus or equipment).

- | | |
|----------|---|
| 13/08/99 | Thermocouple was added in new crosspiece before filter. |
| 17/08/99 | Shifted equipment (desk mounted, more compact) to Cooper's Brewery Ltd, Statenborough St., Leabrook SA. |
| 26/08/99 | New T-piece was fitted. V6 and associated fill line were added to the pilot plant. |
| 30/08/99 | P-gauge above filter replaced with 250 kPa range unit for closer control. |
| 15/02/00 | Leak in the tubing from N ₂ vessel was fixed. |
| 14/04/00 | Shifted equipment back to Chemical Engineering Department, Adelaide University. |

APPENDIX C: Results of preliminary trials

Microbial Analysis

The following media were used to filter a yeast solution (beer simulant) in the pilot plant. Approximately 30 – 35 g of media were used in each test and using 206 kPa (30 psig).

- milled synthetic zeolite (63-125 μm) [“Phonosorb”]
- milled synthetic zeolite(125-250 μm) [“Phonosorb”]
- diatomaceous earth [“Celite 503”]
- cellulose (cotton wool)
- perlite
- precipitated silica
- pumice
- filter sand

Preparation of the Yeast Solution (Beer Simulant):

Yeast solution was prepared using dry yeast (Wander Premium yeast) used in home brewing that was purchased at a local grocery. One packet containing 5 g of yeast and 200 g sugar were dissolved in lukewarm water and then diluted to 1 L. The solution was then incubated for 24-48 h in a 37⁰C incubator. It was harvested by successive centrifugation and washing with saline solution. The final product was suspended in saline solution and refrigerated until the experimental run. Six batches were prepared.

Cell Count

This method is used to determine the total cell count of the samples.

The following equipment is needed: microscope, hemocytometer, coverslip, flask/vial, pipettor and tips, handheld counter.

The vial of sample is swirled thoroughly to mix the solution and to remove the gas from the solution. The coverslip is placed over the counting area of the hemocytometer. Using the pipettor, approximately 10 μL of the sample (or until the counting area is covered with the sample) is purged into the V-shaped groove. Both the upper and lower grooves can be used for counting. The sample is viewed under the microscope using 10X objective lens first to focus the counting area then shifted to 40X to frame up the counting area of one of the 25 large squares.

Viability Tests:

Two procedures were used for determining the viable cells:

Staining with methylene blue: this method is the same as the hemocytometer test for total cell count except that the sample is stained with methylene blue (.01%). In a 2 ml degassed sample, about 10 drops of methylene blue was added until the sample was dark. It was mixed and allowed to stand for 3-5 minutes. Then it is analyzed using the microscope accordingly. The dead cells are blue.

Plate Count: A modified Savaud's agar was used in this analysis. In a freshly prepared agar plate, 100 μL of sample is carefully transferred to the plate and then spread all over the agar using a triangular spreader. It is then incubated at 27 $^{\circ}\text{C}$ for 2-4 days and then cell colony growth is counted.

A result summary is presented as Table 4.5 of Section 4.2.

Appendix D: Safety issues with zeolite-A material

As emphasised in previous studies¹:

- There is a very remote chance of aluminum leaching from zeolite-A. Aluminum makes up part of the crystalline structure of zeolite. Sodium, potassium or calcium participates in ion exchange during chemical reactions.
- Zeolite-A is the most common product of synthesis because of its relatively smaller cages. This is the most kinetically favourable structure.
- Pores within zeolite-A structure are 100 times smaller than those in DE. The pore size of DE is in the order of one micrometer while zeolite is in the nanometre range. Smaller pores may be more desirable because larger pores may lead to organic chemical reactions as it may provide a space for the reaction to occur within. In this case, zeolite acts as a catalyst to these organic reactions.
- If electron micrographs of the DE and zeolite-A are taken before and after filtration, different organic reactions that may take place may be visible.
- Zeolite-A is non-toxic via oral, dermal, ocular and respiratory exposures.

¹ Information from Dr. Allan Pring, Principal Curator of Minerals and Meteorites, Division of Natural Science, SA Museum, as reported by Davey, Kadir and Pecanek (1997) and adapted from Zeolyst Homepage: <http://www.zeolyst.com/html/faq.html>.

Appendix E: Material and safety data sheets (*MSDS*'s)

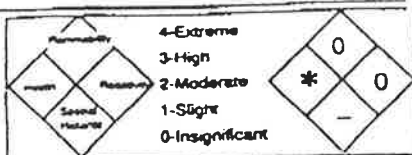
FILCHEM AUSTRALIA PTY. LTD
PO Box H 168, Harris Park, 2150
Phone (02) 9689 2722
Emergency Phone. 1 800 359 434

Material Safety Data Sheet

No.: 2300

Rev. No.: 2

Date Revised: 6/3/97



NFPA FIRE HAZARD SYMBOL
See NFPA 704 for detailed explanation.
* See Section 3 HAZARD IDENTIFICATION

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Trade Name(s): (CELITE® = C) C3Z, C201, C270, C271, C350, C505, C507, C512, C577, FILTER CEL®, STANDARD SUPER CEL®, SSC, X-3

Generic Name: CALCINED DIATOMACEOUS EARTH

Chemical Name: SILICA

Manufacturer: CELITE CORPORATION

Address: P.O. BOX 519

City: LOMPOC

State: CA

Zip: 93438

CAS: 91053-39-3

EINECS: 293-303-4

Formula: SiO₂

Telephone: (805) 735-7791

Emergency: CHEMTREC - USA: (800) 424-9300

International: (703) 527-3887 (collect)

2. COMPOSITION/INFORMATION ON INGREDIENTS

INGREDIENT NAME:	CAS NUMBER:	%	PEL AND TLV (except as noted)
CALCINED DIATOMACEOUS EARTH (DE)	91053-39-3	100	
THIS PRODUCT MAY CONTAIN UP TO 40% CRYSTALLINE SILICA:			
CRISTOBALITE	14464-46-1	< 35	.05mg/M ³ RESPIRABLE CRISTOBALITE, OSHA
QUARTZ	14808-60-7	< 5	.1mg/M ³ RESPIRABLE QUARTZ, OSHA

3. HAZARD IDENTIFICATION

Summary: THIS PRODUCT CONTAINS CRYSTALLINE SILICA (CS), WHICH IS CONSIDERED A HAZARD BY INHALATION. IARC HAS CLASSIFIED INHALATION OF CS AS CARCINOGENIC FOR HUMANS (GROUP 1). CS IS LISTED BY NTP AS A SUBSTANCE WHICH MAY REASONABLY BE ANTICIPATED TO BE A CARCINOGEN. INHALATION OF CS IS ALSO A KNOWN CAUSE OF SILICOSIS, A NONCANCEROUS LUNG DISEASE.

Medical conditions which may be aggravated: PRE-EXISTING UPPER RESPIRATORY AND LUNG DISEASE SUCH AS, BUT NOT LIMITED TO BRONCHITIS, EMPHYSEMA AND ASTHMA.
Target Organ(s): LUNGS, EYES

Acute Health Effects: TRANSITORY UPPER RESPIRATORY OR EYE IRRITATION.

Chronic Health Effects: INHALATION OF CRYSTALLINE SILICA HAS BEEN CLASSIFIED BY IARC AS CARCINOGENIC FOR HUMANS (GROUP 1). INHALATION OF CRYSTALLINE SILICA IS ALSO A KNOWN CAUSE OF SILICOSIS, A NONCANCEROUS LUNG DISEASE CAUSED BY EXCESSIVE EXPOSURE TO CRYSTALLINE SILICA.

Primary Entry Route(s): INHALATION, DUST CONTACT WITH EYES.

Inhalation: IRRITATION AND SORENESS IN THROAT & NOSE. IN EXTREME EXPOSURES SOME CONGESTION MAY OCCUR.

Eyes: TEMPORARY IRRITATION OR INFLAMMATION.

Skin Contact: NA

Skin Absorption: NA

Ingestion: NOT HAZARDOUS WHEN INGESTED.

4. FIRST AID MEASURES

Inhalation: REMOVE TO FRESH AIR. DRINK WATER TO CLEAR THROAT AND BLOW NOSE TO EVACUATE DUST.

Eyes: FLUSH EYES WITH LARGE QUANTITIES OF WATER. IF IRRITATION PERSISTS CONSULT A PHYSICIAN.

Skin Contact: NA

Skin Absorption: NA

Ingestion: NA

5. FIRE FIGHTING MEASURES

Flash Point (Method): NONFLAMMABLE

Flammable Limits: LEL: NA

UEL: NA

Extinguishing Media: NA

Unusual Fire or Explosion Hazards: NONE

NFPA Flammable/Combustible Liquid Classification: NA

Auto-Ignition Temperature: NA

Special Fire-Fighting Procedures: NONE

6. ACCIDENTAL RELEASE MEASURES

Procedures for Spill/Leak: VACUUM CLEAN DUST WITH EQUIPMENT FITTED WITH HEPA FILTER. USE A DUST SUPPRESSANT SUCH AS WATER IF SWEEPING IS NECESSARY.

7. HANDLING AND STORAGE

MINIMIZE DUST GENERATION AND ACCUMULATION. AVOID BREATHING DUST, AVOID CONTACT WITH EYES. SEAL BROKEN BAGS IMMEDIATELY. CONTINUE TO FOLLOW ALL MSDS/LABEL WARNINGS WHEN HANDLING EMPTY CONTAINERS.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Goggles: GOGGLES OR SAFETY GLASSES WITH SIDESHIELDS ARE RECOMMENDED.

Gloves: NOT NORMALLY REQUIRED.

Respirator: <10X PEL, USE 3M 9900; <100X PEL, USE MSA ULTRA-TWIN WITH H FILTER; <200X PEL, USE MSA 01-00-06 WITH TYPE C SUPPLIED AIR UNIT (CONT. FLOW MODE); OR EQUIVALENT.

Ventilation: USE SUFFICIENT NATURAL OR MECHANICAL VENTILATION TO KEEP DUST LEVEL BELOW PEL.

Other: Special Considerations for repair/maintenance of contaminated equipment: INSURE PROPER RESPIRATORY PROTECTION.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance and Odor: FINE PINK POWDER, NO ODOR.

Boiling Point: NA

Evaporation Rate (= 1): NA

Specific Gravity (water = 1) : 2.3

Vapor Pressure: NA

Melting Point: NA

% Volatile by Volume: NIL

Water Solubility (%): NEGLIGIBLE

Vapor Density (Air=1): NA

pH: 6 - 8

10. STABILITY AND REACTIVITY

MATERIAL IS STABLE.

HAZARDOUS POLYMERIZATION CANNOT OCCUR.

Chemical Incompatibilities: HYDROFLUORIC ACID.

Conditions to Avoid: NONE IN DESIGNED USE.

11. TOXICOLOGICAL INFORMATION

Summary: THIS PRODUCT CONTAINS CRYSTALLINE SILICA (CS), WHICH IS CONSIDERED A HAZARD BY INHALATION. IARC HAS CLASSIFIED CS AS CARCINOGENIC FOR HUMANS (GROUP 1). CS IS LISTED BY NTP AS A SUBSTANCE WHICH MAY REASONABLY BE ANTICIPATED TO BE A CARCINOGEN. CS IS ALSO A KNOWN CAUSE OF SILICOSIS, A NONCANCEROUS LUNG DISEASE.

12. ECOLOGICAL INFORMATION

GENERALLY CONSIDERED CHEMICALLY INERT IN THE ENVIRONMENT. USED MATERIAL WHICH HAS BECOME CONTAMINATED MAY HAVE SIGNIFICANTLY DIFFERENT CHARACTERISTICS BASED ON THE CONTAMINANT AND SHOULD BE EVALUATED ACCORDINGLY.

13. DISPOSAL CONSIDERATIONS

WASTE IS NOT HAZARDOUS AS DEFINED BY RCRA (40 CFR 261). METHOD OF DISPOSAL IS TO LANDFILL. OTHER STATE AND LOCAL REGULATIONS MAY VARY, CONSULT LOCAL AGENCIES AS NEEDED. USED MATERIAL WHICH HAS BECOME CONTAMINATED MAY HAVE SIGNIFICANTLY DIFFERENT CHARACTERISTICS BASED ON THE CONTAMINANTS AND SHOULD BE EVALUATED ACCORDINGLY.

14. TRANSPORTATION INFORMATION

D.O.T. Proper Shipping Name: EARTHP, DIATOMACEOUS, CRUDE OR GROUND Hazard Classification: NOT CLASSIFIED
Reportable Quantities: NOT APPLICABLE UN (United Nations), NA (North America) Number: NOT APPLICABLE

15. REGULATORY INFORMATION

OSHA Hazard Communications Standard, 29 CFR 1910.1200: MATERIAL IS CONSIDERED HAZARDOUS. SEE SECTION 3 OF THIS MSDS.
RCRA: THIS MATERIAL IS NOT DEFINED AS HAZARDOUS WASTE PER 40 CFR 261.
TSCA: THIS MATERIAL IS LISTED IN THE TSCA INVENTORY. AND IS NOT OTHERWISE REGULATED BY TSCA SEC. 4, 5, 6, 7 OR 12.
CERCLA: MATERIAL IS NOT REPORTABLE UNDER CERCLA. LOCAL REQUIREMENTS MAY VARY.
SARA: 311/312 HAZARD CATEGORIES -IMMEDIATE AND DELAYED HEALTH, 313 REPORTABLE INGREDIENTS - NONE.
California Proposition 65: THIS PRODUCT CONTAINS CHEMICALS KNOWN TO THE STATE OF CALIFORNIA TO CAUSE CANCER.

16. OTHER INFORMATION

Storage Segregation Hazard Classes: NA

Special Handling/Storage: REPAIR ALL BROKEN BAGS IMMEDIATELY.

Special Workplace Engineering Controls: ADEQUATE VENTILATION TO KEEP DUST LEVEL BELOW PEL

Prepared/Revised by: CHRIS PAULEY

Title: DIRECTOR, HEALTH & SAFETY SERVICES

As of the date of preparation of this document, the foregoing information is believed to be accurate and is provided in good faith to comply with applicable federal and state law(s). However, no warranty or representation with respect to such information is intended or given.

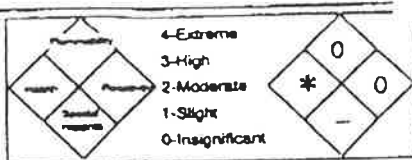
Material Safety Data Sheet



No.: 2776

Rev. No.: 2

Date Revised: 6/3/97



NFPA FIRE HAZARD SYMBOL
See NFPA 704 for detailed explanation.
* See Section 3 HAZARD IDENTIFICATION

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Trade Name(s): FIBRA-CEL® - DIATOMACEOUS EARTH CELLULOSE FIBER BLENDS

Generic Name: FLUX CALCINED DIATOMACEOUS EARTH
Chemical Name: SILICA /CELLULOSE BLEND
Manufacturer: CELITE CORPORATION
Address: P.O. BOX 519
City: LOMPOC State: CA Zip: 93438

CAS: FC DE - 68855-54-9, CELLULOSE - 9004-34-6
EINECS: FC DE - 272-488-0, CELLULOSE - UNKNOWN
Formula: $\text{SiO}_2/(\text{C}_6\text{H}_{10}\text{O}_5)_n$
Telephone: (805) 735-7791
Emergency: CHEMTREC - USA: (800) 424-9300
International: (703) 527-3887 (collect)

2. COMPOSITION/INFORMATION ON INGREDIENTS

INGREDIENT NAME:	CAS NUMBER:	%	PEL AND TLV
FLUX CALCINED DIATOMACEOUS EARTH (DE)	68855-54-9	85-95	SEE BELOW
CELLULOSE FIBER	9004-34-6	5-15	5.mg/M ³ RESPIRABLE NUISANCE DUST, OSHA 10 mg/M ³ TOTAL NUISANCE DUST, ACGIH
THIS PRODUCT MAY CONTAIN UP TO 75% CRYSTALLINE SILICA:			
CRISTOBAULITE	14464-46-1	< 70	.05 mg/M ³ RESPIRABLE CRISTOBAULITE, OSHA
QUARTZ	14808-60-7	< 5	.1mg/M ³ RESPIRABLE QUARTZ, OSHA

3. HAZARD IDENTIFICATION

Summary: THIS PRODUCT CONTAINS CRYSTALLINE SILICA (CS), WHICH IS CONSIDERED A HAZARD BY INHALATION. IARC HAS CLASSIFIED INHALATION OF CS AS CARCINOGENIC FOR HUMANS (GROUP 1). CS IS LISTED BY NTP AS A SUBSTANCE WHICH MAY REASONABLY BE ANTICIPATED TO BE A CARCINOGEN. INHALATION OF CS IS ALSO A KNOWN CAUSE OF SILICOSIS, A NONCANCEROUS LUNG DISEASE.

Medical conditions which may be aggravated: PRE-EXISTING UPPER RESPIRATORY AND LUNG DISEASE SUCH AS, BUT NOT LIMITED TO BRONCHITIS, EMPHYSEMA AND ASTHMA.
Target Organ(s) : LUNGS, EYES

Acute Health Effects: TRANSITORY UPPER RESPIRATORY OR EYE IRRITATION.

Chronic Health Effects: INHALATION OF CRYSTALLINE SILICA HAS BEEN CLASSIFIED BY IARC AS CARCINOGENIC FOR HUMANS (GROUP 1). INHALATION OF CRYSTALLINE SILICA IS ALSO A KNOWN CAUSE OF SILICOSIS, A NONCANCEROUS LUNG DISEASE CAUSED BY EXCESSIVE EXPOSURE TO CRYSTALLINE SILICA.

Primary Entry Route(s) : INHALATION, DUST CONTACT WITH EYES.

Inhalation: IRRITATION AND SORENESS IN THROAT & NOSE. IN EXTREME EXPOSURES SOME CONGESTION MAY OCCUR.

Eyes: TEMPORARY IRRITATION OR INFLAMMATION.

Skin Contact: NA Skin Absorption: NA Ingestion: NOT HAZARDOUS WHEN INGESTED.

4. FIRST AID MEASURES

Inhalation: REMOVE TO FRESH AIR. DRINK WATER TO CLEAR THROAT AND BLOW NOSE TO EVACUATE DUST.

Eyes: FLUSH EYES WITH LARGE QUANTITIES OF WATER. IF IRRITATION PERSISTS CONSULT A PHYSICIAN.

Skin Contact: NA Skin Absorption: NA Ingestion: NA

5. FIRE FIGHTING MEASURES

Flash Point (Method) : NONFLAMMABLE

NFPA Flammable/Combustible Liquid Classification: NA

Flammable Limits: LEL: NA

UEL: NA

Auto-Ignition Temperature: NA

Extinguishing Media: NA

Unusual Fire or Explosion Hazards: NONE

Special Fire-Fighting Procedures: NONE

6. ACCIDENTAL RELEASE MEASURES

Procedures for Spill/Leak: VACUUM CLEAN DUST WITH EQUIPMENT FITTED WITH HEPA FILTER. USE A DUST SUPPRESSANT SUCH AS WATER IF SWEEPING IS NECESSARY.

7. HANDLING AND STORAGE

MINIMIZE DUST GENERATION AND ACCUMULATION. AVOID BREATHING DUST, AVOID CONTACT WITH EYES. SEAL BROKEN BAGS IMMEDIATELY. CONTINUE TO FOLLOW ALL MSDS/LABEL WARNINGS WHEN HANDLING EMPTY CONTAINERS.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Goggles: GOGGLES OR SAFETY GLASSES WITH SIDESHIELDS ARE RECOMMENDED.

Gloves: NOT NORMALLY REQUIRED.

Respirator: <10X PEL USE 3M 9900; <100X PEL USE MSA ULTRA-TWIN WITH H FILTER; <200X PEL USE MSA 01-00-06 WITH TYPE C SUPPLIED AIR UNIT (CONT. FLOW MODE); OR EQUIVALENT.

Ventilation: USE SUFFICIENT NATURAL OR MECHANICAL VENTILATION TO KEEP DUST LEVEL BELOW PEL.

Other: Special Considerations for repair/maintenance of contaminated equipment: INSURE PROPER RESPIRATORY PROTECTION.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance and Odor: FINE WHITE/TAN POWDER, NO ODOR.

Boiling Point: NA

Evaporation Rate (= 1): NA

Specific Gravity (water = 1): 2.3

Vapor Pressure: NA

Melting Point: NO

% Volatile by Volume: NIL

Water Solubility (%): NEGLIGIBLE

Vapor Density (Air=1): NA

pH: ND

10. STABILITY AND REACTIVITY

MATERIAL IS STABLE.

HAZARDOUS POLYMERIZATION CANNOT OCCUR.

Chemical Incompatibilities: HYDROFLUORIC ACID.

Conditions to Avoid: NONE IN DESIGNED USE.

11. TOXICOLOGICAL INFORMATION

Summary: THIS PRODUCT CONTAINS CRYSTALLINE SILICA (CS), WHICH IS CONSIDERED A HAZARD BY INHALATION. IARC HAS CLASSIFIED CS AS CARCINOGENIC FOR HUMANS (GROUP 1). CS IS LISTED BY NTP AS A SUBSTANCE WHICH MAY REASONABLY BE ANTICIPATED TO BE A CARCINOGEN. CS IS ALSO A KNOWN CAUSE OF SILICOSIS, A NONCANCEROUS LUNG DISEASE.

12. ECOLOGICAL INFORMATION

GENERALLY CONSIDERED CHEMICALLY INERT IN THE ENVIRONMENT. USED MATERIAL WHICH HAS BECOME CONTAMINATED MAY HAVE SIGNIFICANTLY DIFFERENT CHARACTERISTICS BASED ON THE CONTAMINANT AND SHOULD BE EVALUATED ACCORDINGLY.

13. DISPOSAL CONSIDERATIONS

WASTE IS NOT HAZARDOUS AS DEFINED BY RCRA (40 CFR 261). METHOD OF DISPOSAL IS TO LANDFILL. OTHER STATE AND LOCAL REGULATIONS MAY VARY, CONSULT LOCAL AGENCIES AS NEEDED. USED MATERIAL WHICH HAS BECOME CONTAMINATED MAY HAVE SIGNIFICANTLY DIFFERENT CHARACTERISTICS BASED ON THE CONTAMINANTS AND SHOULD BE EVALUATED ACCORDINGLY.

14. TRANSPORTATION INFORMATION

D.O.T. Proper Shipping Name: EARTH, DIATOMACEOUS, CRUDE OR GROUND **Hazard Classification:** NOT CLASSIFIED
Reportable Quantities: NOT APPLICABLE **UN (United Nations), NA (North America) Number:** NOT APPLICABLE

15. REGULATORY INFORMATION

OSHA Hazard Communications Standard, 29 CFR 1910.1200: MATERIAL IS CONSIDERED HAZARDOUS, SEE SECTION 3 OF THIS MSDS.

RCRA: THIS MATERIAL IS NOT DEFINED AS HAZARDOUS WASTE PER 40 CFR 261.

TSCA: THIS MATERIAL IS LISTED IN THE TSCA INVENTORY, AND IS NOT OTHERWISE REGULATED BY TSCA SEC. 4, 5, 6, 7 OR 12.

CERCLA: MATERIAL IS NOT REPORTABLE UNDER CERCLA. LOCAL REQUIREMENTS MAY VARY.

SARA: 311/312 HAZARD CATEGORIES -IMMEDIATE AND DELAYED HEALTH, 313 REPORTABLE INGREDIENTS - NONE.

California Proposition 65: THIS PRODUCT CONTAINS CHEMICALS KNOWN TO THE STATE OF CALIFORNIA TO CAUSE CANCER.

16. OTHER INFORMATION

Storage Segregation Hazard Classes: NA

Special Handling/Storage: REPAIR ALL BROKEN BAGS IMMEDIATELY.

Special Workplace Engineering Controls: ADEQUATE VENTILATION TO KEEP DUST LEVEL BELOW PEL

Prepared/Revised by: CHRIS PAULEY

Title: DIRECTOR, HEALTH & SAFETY SERVICES

As of the date of preparation of this document, the foregoing information is believed to be accurate and is provided in good faith to comply with applicable federal and state laws. However, no representation with respect to such information is intended or given.

MATERIAL SAFETY DATA SHEET

CORPORATE RESEARCH & DEVELOPMENT

SCHENECTADY, N. Y. 12305

MATERIALS INFORMATION SERVICES

No. 69

NATURAL DIATOMACEOUS EARTH

Date September 1980

SECTION I. MATERIAL IDENTIFICATION

Reviewed: February 1982

MATERIAL NAME: NATURAL DIATOMACEOUS EARTH

DESCRIPTION: Submicroscopic siliceous skeletons of prehistoric diatoms.

OTHER DESIGNATIONS: Diatomite, Amorphous Silica, Kieselguhr, Silicon Dioxide, SiO₂, GE Materials D4E13 and D4E16 (natural grades), CAS #061 790 532

MANUFACTURER: Available from several suppliers, including:
 Johns-Manville Sales Corp., Ken Caryl Ranch Grefco, Inc., 3450 Wilshire Blvd.
 Denver, CO 80202 Phone: (303) 979-1000 Los Angeles, CA Phone: (213) 381-5081
 CELITE (Natural Grades) DICALITE (Natural Grades)

SECTION II. INGREDIENTS AND HAZARDS

				%	HAZARD DATA
Diatomaceous Earth (Natural) with ca 4% free moisture* 8-hr TWA**				ca 100	8-hr TWA 1.5 mg/m ³ ** (Respirable Mass)
Composition (SiO ₂)	%	Resp. Mass	Total Mass		
Amorphous Silica	>95	3 mg/m ³	6 mg/m ³		
Quartz	< 5	0.1	0.3		
Cristobalite	Trace	0.05	0.15		
Tridymite	Trace	0.05	0.15		

*May contain small amounts of oxides of potassium, calcium, aluminum and iron, depending on ore source; 3-4% combined H₂O

**ACGIH 1981 TLV. The current OSHA standard for amorphous silica and natural diatomaceous earth is 20 mppcf or 80% quartz content mg/m³.

Rat, Oral
LD₅₀ 3160 mg/kg

SECTION III. PHYSICAL DATA

Boiling point, at 760 mm Hg, deg C - 2230 Softening point, deg C ----- 1427
 Solubility in water ----- insoluble Melting point, deg C ----- 1710
 Density ----- 2.2 Molecular weight ----- 60.09
 Particle size, microns ----- <10 dependent on treatment (as SiO₂)

Appearance & Odor: Light gray or buff colored powder (also supplied in the form of blocks or bricks); odorless.

SECTION IV. FIRE AND EXPLOSION DATA

Flash Point and Method	Autoignition Temp.	Flammability Limits In Air	LOWER	UPPER
N/A				

Extinguishing Media: Use media appropriate to surrounding fire.
 This material is noncombustible, but avoid generating airborne dust.
 When heated to extreme temperatures, it can crystallize or melt into a glass.
 Firefighters may need respiratory protection under dusty conditions.

SECTION V. REACTIVITY DATA

Material is stable under ordinary circumstances. Does not polymerize.
 Reacts with hydrofluoric acid to produce toxic silicon tetrafluoride gas and with xenon hexafluoride to produce explosive xenon trioxide. Heating with alkali carbonates can produce a vigorous reaction; when wet and heated with Mg, it can explode. It can react exothermally with oxygen difluoride and explosively with chlorine trifluoride.
 It can absorb up to 4 times its weight in water, and when finely divided, is soluble in strong or molten alkalis.
 When heated to high temperature, as in calcining (especially in the presence of alkaline flux), this material forms crystalline silicas, cristobalite and tridymite, both of which are very active in causing silicosis when inhaled.

GENERAL ELECTRIC

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SECTION VI. HEALTH HAZARD INFORMATION

TLV 1.5 mg/m³ (See Sect. II)

Natural Diatomaceous Earth presents a moderate risk from inhalation of particulate, otherwise it is nontoxic and relatively harmless.* Pneumoconiosis is a possible occupational hazard from excessive inhalation. Material can be drying and abrasive to skin.

FIRST AID:

Eye Contact: Flush eyes thoroughly with water to remove particulate. If irritation persists, get medical attention.

Skin Contact: Remove by washing with soap and water.

Inhalation: Remove to fresh air. Restore and/or support breathing as required. Get medical attention.

*Heating at high temperature (calcining at 900 C) transforms the relatively benign amorphous silica to crystalline forms which can be much more active in producing silicosis in the lungs.

SECTION VII. SPILL, LEAK, AND DISPOSAL PROCEDURES

Notify safety personnel of major spills. Provide ventilation. Provide clean up personnel with protection against eye contact and inhalation of dust.

Pick up small spills taking care to avoid raising dust clouds (use vacuum or wet sweeping). Place in a closed container for disposal.

DISPOSAL: Use waste containers suitable for transportation and dispose in approved landfill. Follow Federal, State and Local regulations.

SECTION VIII. SPECIAL PROTECTION INFORMATION

Provide adequate general and local ventilation to meet TLV requirements. Provide workers with dust respirators for use in emergency or nonroutine situations where dust levels may exceed the TLV.

Workers should wear safety goggles or face shield and thick work gloves.

Eyewash fountains should be provided in areas of use and handling.

Preclude from exposure those individuals with pulmonary disease.

SECTION IX. SPECIAL PRECAUTIONS AND COMMENTS

Store in closed containers in a clean, dry, well-ventilated area. Handle in a manner which will avoid generation of dust.

Use good housekeeping practices to prevent accumulation of dust and follow cleaning techniques (vacuuming and wet sweeping) that will keep airborne particulate at a minimum.

Avoid inhalation of dust. Keep out of the eyes. Use with good ventilation.

Calcined grades of diatomaceous earth are much more hazardous when inhaled than is the natural or powdered natural material. See F.R. Dutra, Arch Environ Health, Vol. 11 (Nov. 1965), pp 613-619.

DATA SOURCE(S) CODE: 1-14, 24, 25, 27, 37

Judgments as to the suitability of information herein for purchaser's purposes are necessarily purchaser's responsibility. Therefore, although reasonable care has been taken in the preparation of such information, General Electric Company extends no warranties, makes no representations and assumes no responsibility as to the accuracy or suitability of such information for application to purchaser's intended purposes or for consequences of its use.

APPROVALS: MIS
CRD

Industrial Hygiene
and Safety

MEDICAL REVIEW: 13 Sept. 1980

GENERAL  ELECTRIC

ZEOCHEM

Chemie Uetikon
and United Catalysts Inc.
Joint Venture

P.O. Box 35940
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M A T E R I A L S A F E T Y D A T A S H E E T

I. PRODUCT IDENTIFICATION

PRODUCT Z3-01, 02, 03, 04; Z4-01, 02; Z5-01, 02, 04; Z10-01;
Molecular Sieve 3A-Z8, 3A-Z8-02, 4A-Z8, 5A-Z8, 13X-Z8

FORMULA $Mx/n[AlO_2]x(SiO_2)y + wH_2O$

CHEMICAL NAME Synthetic Sodium Potassium or
Calcium Aluminosilicate

CHEMICAL FAMILY Molecular Sieve
Zeolite

II. (A) INGREDIENTS

<u>COMPONENT</u>	<u>CAS No.</u>	<u>Zeolite Type</u>
Zeolite, NaA	1344-00-9	4A
Zeolite, KA	12736-96-8	3A
Zeolite, CaA	1344-01-0	5A
Zeolite, NaX	1344-00-9	13X
Mg Aluminosilicate	1327-43-1	Clay

II. (B) PRODUCT ANALYSES & EXPOSURE LIMITS

<u>COMPONENT</u>	<u>CAS NO.</u>	<u>%</u>	<u>OSHA/PEL</u>	<u>ACGIH/TLV</u>
Zeolite	See above	75-85	10mg/m ³	10mg/m ³
Mg Aluminosilicate	1327-43-1	23-15	10mg/m ³	10mg/m ³
Quartz	14808-60-7	2-0	0.1mg/m ³	0.1mg/m ³

III. PHYSICAL DATA

MELTING POINT °F >2900

BULK DENSITY 0.68 g/cc

MELTING POINT °C >1600

**PERCENT VOLATILES
BY WEIGHT** <5%

DATE OF ISSUE: January 1, 1986

DATE OF REVISION: August 29, 1990

PAGE 1

PRODUCT	Z3-01, 02, 03, 04; Z4-01, 02; Z5-01, 02; Z10-01; Molecular Sieve 3A-Z8, 3A-Z8-02, 4A-Z8, 5A-Z8, 13X-Z8
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APPEARANCE AND ODOR	Product may appear as light tan bead, cake or powder.
------------------------	---

IV. FIRE AND EXPLOSION HAZARD DATA

FLASH POINT	Nonflammable	FIREFIGHTING MEDIA	Dry chemical, water spray or foam.
--------------------	--------------	---------------------------	------------------------------------

FIRE AND EXPLOSION HAZARD - Negligible fire and explosion hazard when exposed to heat or flame by reaction with incompatible substances.

FIREFIGHTING - Nonflammable solids, liquids or gases: Cool containers that are exposed to flames with water from the side until well after fire is out. For massive fire in enclosed area, use unmanned hose holder or monitor nozzles; if this is impossible, withdraw from area and let fire burn. Withdraw immediately in case of rising sound from venting safety device or discoloration of the tank due to fire.

V. HEALTH HAZARD DATA

Health hazards may arise from ingestion, inhalation and contact with the skin and eyes. Ingestion may result in damage to throat, esophagus, and/or gastro-intestinal tract. Inhalation may cause burning of the upper respiratory tract and/or temporary or permanent lung damage. Prolonged or repeated contact with the skin, in the absence of proper hygiene, may cause dryness, irritation, and/or dermatitis. Contact with eye tissue may result in irritation, burns or conjunctivitis. This product contains a small amount of crystalline silica which may cause delayed respiratory disease if inhaled over a prolonged period of time. IARC Monographs on the evaluation of the Carcinogenic Risk of Chemicals to Humans (volume 42, 1987) concludes that there is "limited evidence" of the carcinogenicity of crystalline silica to humans. IARC classification 2A.

First Aid (Inhalation) - Remove to fresh air immediately. If breathing has stopped, give artificial respiration. Keep affected person warm and at rest. Get medical attention immediately.

First Aid (Ingestion) - If large amounts have been ingested, give emetics to cause vomiting. Stomach siphon may be applied as well. Milk and fatty acids should be avoided. Get medical attention immediately.

PRODUCT Z3-01, 02, 03, 04; Z4-01, 02; Z5-01, 02; Z10-01;
Molecular Sieve 3A-Z8, 3A-Z8-02, 4A-Z8, 5A-Z8, 13X-Z8

First Aid (Eyes) - Wash affected areas immediately and carefully for 15 to 20 minutes with running water. Get prompt medical attention.

First Aid (Skin) - Wash with soap and water.

NOTE TO PHYSICIAN - This product is a desiccant and generates heat as it absorbs water. The used product can contain material of hazardous nature. Identify that material and treat accordingly.

VI. REACTIVITY DATA

Reactivity - Is stable under normal temperatures and pressures in sealed containers. Hazardous polymerization will not occur. Moisture can cause rise in temperature which may result in burn. Avoid sudden contact with high concentrations of chemicals having high heats of adsorption such as olefins, HCl, etc.

VII. SPILLS OR LEAK PROCEDURES

Notify safety personnel of spills or leaks. Cleanup personnel need protection against inhalation of dusts or fumes. Eye protection is required. Vacuuming or wet methods of cleanup are preferred. Place in appropriate containers for disposal keeping airborne particulate at a minimum.

Disposal Method - In selecting the method of disposal, applicable local, state and federal regulations should be consulted.

VIII. SPECIAL PROTECTION INFORMATION

Respiratory Protection - Provide a NIOSH/MSHA jointly approved respirator in the absence of proper environmental control or where TLV for crystalline silica may be exceeded. Contact your safety equipment supplier for proper mask type.

Ventilation - Provide general and/or local exhaust ventilation to keep exposures below the threshold limit value. Ventilation used must be designed to prevent spots of dust accumulation or recycling of dusts.

Protective Clothing - Wear protective clothing, including gloves, to prevent repeated or prolonged skin contact.

PRODUCT Z3-01, 02, 03, 04; Z4-01, 02; Z5-01, 02; Z10-01;
 Molecular Sieve 3A-Z8, 3A-Z8-02, 4A-Z8, 5A-Z8, 13X-Z8

Eye Protection - Chemical splash goggles designed in compliance with OSHA regulations are recommended. Consult your safety equipment supplier.

IX. REGULATORY INFORMATION

The information presented herein is believed to be accurate but is not warranted. Recipients are advised to confirm in advance that the information is current and applicable to meet their circumstances.

This product contains substances which appear on lists of the indicated act or agency.

XX American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Values for Chemical Substance in the Work Environment

XX California Proposition 65

_____ Clean Air Act 40 CFR 61

_____ Clean Water Act 40 CFR 116

_____ Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) 40 CFR 302

XX International Agency for Research on Cancer (IARC) Monographs on the Evaluation of Carcinogenic Risks to Humans Volumes 1-42

_____ NTP Annual Report on Carcinogens

XX Occupational Safety and Health Administration (OSHA) 29 CFR 1910

_____ Resource Conservation and Recovery Act (RCRA) 40 CFR 261 Subpart C

_____ Superfund Amendments and Reauthorization Act of 1986 (SARA) Title III Section 313 40 CFR 372

XX Toxic Substances Control Act (TSCA) 40 CFR 700



Celite Corporation
P O Box 519
Lompoc, California 93438-0519
Telephone: (805) 735-7791

Technical Data

STANDARD SUPER-CEL®

TYPICAL PHYSICAL PROPERTIES

Color	Buff/Pink
Appearance	Powder
Origin	Plankton Marine Diatomite
Description	Calcined Filter Aid
Median Pore Size, Microns	3.5
Permeability, D'Arcy's	0.3
Wet Density (lbs/ft ³)	19.0
150 Mesh Screen Analysis % Retained	3.0
Moisture, as shipped, %	0.5
Water Solubies %	0.10
Median Particle Size, microns	15.0
Specific Gravity	2.2

TYPICAL CHEMICAL ANALYSIS, %

Ignition Loss	0.5
SiO ₂	91.1
Al ₂ O ₃	4.0
Fe ₂ O ₃	1.3
P ₂ O ₅	0.2
TiO ₂	0.2
CaO	0.5
MgO	0.6
Na ₂ O + K ₂ O	1.1
pH	7.0

The typical physical or chemical properties of Celite Corporation products represent average values obtained in accordance with accepted test methods and are subject to normal manufacturing variations. They are supplied as a technical service and are subject to change without notice. Typical data shown above are considered accurate and reliable; however, no guarantee is given or intended.

Celite

P.O. Box 519
Lompoc, CA 93438-0519
Telephone: 805 735-7791

Specification Data

EFFECTIVE: 04/10/89 • SUPERCEDES 09/26/85

Page 1 of 1

FIBRA-CEL® 5F

Description: Precoat Filter Aid consisting of diatomite and cellulose

Constituents: Target Value: 85.0% Standard Super Cel
15.0% Fibra-Cel SW10

Specification Range: $\pm 1.25\%$

Each specification to which any of our filter aids is made primarily relates to the flow rate and clarifying properties as measured under standard conditions in special equipment. Any product which meets the test described above is considered by us as specification material, since the product is sold to perform a certain specific filtration function, and that test is a measure of this function.

For important health and safety information, please refer to MSDS

Celite Corporation

The typical physical or chemical properties of Celite Corporation products represent average values obtained in accordance with accepted test methods and are subject to normal manufacturing variations. They are supplied as a technical service and are subject to change without notice. Typical data shown above are considered accurate and reliable; however, no guarantee is given or intended.

Molecular Sieve 4A

Description

Molecular Sieve 4A is an alkali aluminosilicate. It is the sodium form of the Molecular Sieve type A and has an effective pore opening of about 4 angstroms (0,4 nm).

Typical Chemical Formula

$\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 2 \text{SiO}_2 \cdot n \text{H}_2\text{O}$

Applications

Molecular Sieve 4A is commonly used for the following applications:

General drying and purification of hydrocarbon fluids, such as natural gas, LPG, air, inert and atmospheric gases; removal of carbon dioxide, ammonia and methanol from fluid streams. Special grades are used in, for example, the drying of refrigerants, and the drying of air in air brakes. Further duties include use as a packaging desiccant. Molecular Sieve 4A will adsorb molecules with a kinetic diameter of less than 0,4 nm and exclude those larger.

Types

4A - 401 is the standard grade. It is used in general drying duties. There are other grades (4A-402, 4A-404 etc.) with particular properties enhanced for dedicated duties, such as 4A-404 especially designed for natural gas dehydration. The properties given below are those for 4A-401. Other properties may also be specified, or changes made, for the other grades. Data sheets on the grades for particular applications are also available. Molecular Sieve 4A is also available in powder form.

Regeneration

Molecular Sieve type 4A can be regenerated by either decreasing the partial pressure of the adsorbate, or increasing the temperature of the molecular sieve. The former method is called pressure swing adsorption (PSA), or vacuum swing adsorption (VSA) if a vacuum is used, while the latter is termed thermal swing adsorption. To remove adsorbed impurities to a useful level a regeneration or purge gas temperature from ambient up to 300°C is required (but not exceeding 450°C).

Typical Properties of Molecular Sieve 4A-401

Chemie Uetikon
Test Method

Nominal pore diameter	4 angstroms (0,4 nm)			
Type of crystal structure	cubic			
Bulk density	740 g/l			Si 5 / 47
Equilibrium water capacity at 20°C/55% r.h.	22% wt.			Si 5 / 49
Water content (as shipped)	1,5% wt. (max.)			Si 5 / 49a
Heat of adsorption (max.)	4200 KJ/kg water			
Specific heat (approx.)	1,07 KJ/kg °C			
Bead sizes (nominal)	2,5–5 mm	2–3 mm	1–2 mm	Si 5 / 41
equivalent (approx.)	4 x 8 mesh	8 x 12 mesh	10 x 18 mesh	
Crush strength	7 kp	4 kp	2 kp	Si 5 / 46

Shipping Information

Molecular Sieve 4A beads are shipped in the following standard packaging:

in JUMBOPAK big-bags of 600 kg and 1000 kg,

in steel drums of 140 kg, 80 kg and 40 kg,

cartons of 25 kg

all with inner polyethylene bags.



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UETIKON

Appendix F: Sensory analyses forms

Form 1

Evaluation of Beer
(Triangular Method)

Name: _____
Date : _____

Product Submitted to Test: Filtered beer using alternative media (KEN)

Problem: Three samples are given to you; please encircle the number of sample that differs from the other two.

SET OF THREE SAMPLES

Comments:

Conducted by:

Maria P.M. Marquez
Department of Chemical Engineering
University of Adelaide
Adelaide, SA 5000

With the assistance of Coopers Brewery
Statenborough Street
LeaBrook, SA 5068

Form 2

Evaluation of Beer Samples

(Descriptive Method)

Sample Code: _____

Please rate the intensity of the following parameters using the scale from 0 to 10
(0 = poor, 10 = excellent)

	rating
Aroma: (fruity, grassy, yeasty, sulfury, oxidized, grainy, etc)	_____
comments: _____	
Color: (too light, too dark)	_____
comments: _____	
Clarity: (cloudy, clear)	_____
comments: _____	
Taste: (bitter, sour, sweet, metallic)	_____
comments: _____	
Drinkability and Overall Impression: (pleasant, unpleasant)	_____
comments: _____	

Name: _____

Date: _____

Conducted by:
 Maria P.M. Marquez
 Department of Chemical Engineering
 University of Adelaide
 Adelaide, SA 5000

With the assistance of Coopers Brewery, Statenborough Street LeaBrook SA 5068

NOTATION

A cross sectional area of filter media (cake) available for filtration

L bed (cake) depth (thickness)

R bed (cake medium) flow resistance

V flow rate of filtrate

Greek Symbols

α bed (cake) specific resistance

ε bed (cake) porosity

μ Viscosity of the filtrate (beer)

ΔP pressure driving force for filtration applied across bed (cake)

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