

8. Carbonising

David Wesmoreland

Learning objectives

On completion of this topic you should be able to:

- developed an understanding of why certain wools need to be carbonised
- an understanding of the basic principles involved in the carbonising process
- an understanding of the parameters to be controlled during the process
- an awareness of the problems which can be encountered and the adverse affect they can have on the final product

Key terms and concepts

Acidising, drying, baking, crushing, de-dusting, neutralisation, vegetable matter, chemical damage.

Introduction to the topic

This topic describes the process of carbonising wool in loose stock form following aqueous wool scouring.

8.1 Carbonising of wool in loose stock form

Introduction

Carbonising is the chemical process which is used to remove vegetable matter (VM) from wool. The VM, which may be seeds, burrs, grass etc., is predominantly made up of cellulose, hemicellulose and lignin whereas the wool is principally protein. The carbonising process makes use of the difference in the stability of proteins and cellulose to the effects of mineral acids. Cellulose is made up of carbon, hydrogen and water with a chemical formula of $C_6H_{10}O_5$. It can be seen from the formula that the hydrogen and oxygen are present in the same ratio as they are in water. Therefore, in considering the underlying principle of carbonising, it may be assumed that all the VM consists of carbon and water in certain proportions. If these two substances are separated the latter can be evaporated away leaving the carbon in a brittle form. The most commonly used acid for this purpose is sulphuric acid. In its simplest form, carbonising follows a traditional wool scouring process and consists of acidification, drying, baking, mechanical crushing and dusting to remove the embrittled VM, neutralisation and final drying. This basic process of loose wool carbonising has remained essentially unaltered for many years.

Over the past few years the trend has been for less wool to be carbonised in the Northern Hemisphere, there are now no carbonising lines still operating in the UK for example. The majority of it now being processed in Australia, South Africa and Asia. Even in Australia the number of raw wool carbonising plants has diminished with only four currently operating, as of June 2004, three of these being in Victoria and one in South Australia. It is possible that the plant at Warwick, in Queensland, may re-open in the near future due to increasing demand. The high fault (i.e. high vegetable-matter content) wools tend to be processed either in the country of origin or near to it.

Carbonising is primarily used for treating card waste, noils and high VM wools which are destined for subsequent processing via the woollen route. Very little processing of wools for worsted production is carried out. Where the VM content is from medium to low, say a maximum of 2-3%, mechanical removal of the VM is usually adequate with the various stages of carding, gilling and combing in the worsted process being sufficient to produce a speck free top. The machinery used in woollen processing generally cannot accommodate medium to high levels of VM. Low to medium levels of VM can be accommodated if the spun wool is to be processed into a fabric which will subsequently be carbonised in the piece.

In the first half of the 20th century a considerable amount of research was carried out by various workers whose prime aim was to minimise the chemical damage imparted to the wool fibre by the acidic conditions. Various alternatives to sulphuric acid were proposed, such as calcium chloride, hydrochloric acid, aluminium chloride and thionyl chloride to name but a few, but commercial operators have, in general, stuck with the sulphuric acid process due to its cheapness and ease of control. During the 70s and 80s some fundamental research was being carried out on such topics as acidising conditions, drying and baking, effect of detergents and rapid test procedures at DWI in Aachen as well as in Australia and these will be briefly discussed. In more recent times, though, this important area of wool processing has seen no new work published.

8.2 Processing stages

The conventional technique of wool carbonising has the following basic steps:

1. Scouring of the raw wool with non-ionic detergent
2. Rinsing
3. Immersion in a long bowl containing 5-7% (w/v) sulphuric acid, 1-2 g/L detergent (Crewther 1995; Pressley 1955) at 20-30°C
4. Double squeezing and/or continuous centrifuging prior to drying
5. Drying at 60-80°C to a low regain
6. Baking at 95-120°C to carbonise the VM
7. Pass through crushing rollers to crush the embrittled VM
8. Convey to a rotating shaker/de-duster to remove the charred VM dust
9. Pass through a neutralising bowl usually containing sodium carbonate
10. Rinsing with a small addition of detergent
11. Bleaching with hydrogen peroxide at approximately pH 5 with formic acid
12. Final drying.

The production rate through the process is usually low, typically 500-600 kg/h for a 1200 mm wide line, since sufficient time must elapse for the VM to adsorb the acid from the bowl and for subsequent baking to embrittle the acidified cellulose.

8.3 Preparation/scouring

The wool has of course to be opened prior to the scouring operation which precedes the acidising unit. It is important that the wool is opened sufficiently to allow for efficient scouring of the wool, but care must be taken if the wool contains many short fibres as these can be lost at this stage and result in a poor yield. Gentle opening then is essential for wools destined for woollen spun yarn. The raw wool is then scoured to remove any wax and dirt to enable the acidifying process to be more effective. In the early part of the 20th century there was a move in Europe to carbonise 'in the grease' as they thought that the wool wax on the fibre would protect it from damage by the acid. In reality the wax tended to become fixed on the fibre making subsequent scouring very difficult.

8.4 Acidising

A great deal of fundamental research has been carried out on the acidising stage (Mozes 1986: mostly aimed at producing maximum penetration of the acid into the vegetable matter whilst keeping the acid concentration of the fibre at a safe level from the point of view of possible damage (WSR 1964) age during subsequent stages and many reviews have been published (Crewther 1995; Mizell, Davis & Oliva 1962; Mozes 1986; Pressley 1955; Simmonds 1954). As a result there have been many suggestions put forward as to the best conditions to employ. Generally, most workers agree on sulphuric acid being the best acid to use, typically at 5% to 7% w/v concentration in the bath, which is usually at 20-30°C. Lower acid strengths have been proposed where total VM removal is not required, and higher water temperatures have also been suggested, but this can lead to excess acid on the fibre, thus causing damage during baking. The effect of acid content on fibre damage has been noted, Table 8.1, by Robinson and Westmoreland (1989). The use of wetting agents in the acidising bowl has been suggested for more rapid wetting of the burrs giving better acid penetration and for prevention of localised concentrations of acid, by spreading, which again can cause damage during baking. The acidising bowl is usually quite long, 9 to 10 m, to allow sufficient immersion time which can be in the order of 3-5 minutes. In old machines the acid bowl would have been made from wood with a lead lining. In modern machines this has been replaced with stainless steel. Acid concentrations and residence times may need to be adjusted to suit the type and nature of the VM present. Wools with a high percentage of hard heads for example will need to be treated at the higher acid concentration to ensure sufficient acid is absorbed by the hard head to facilitate carbonisation. Alternatives to using sulphuric acid have been tried over the years but none have received any degree of commercial acceptance. Some of these have included aluminium chloride (Joly's process), magnesium and calcium chlorides, hydrochloric acid (as vapour), sodium hydrogen sulphate (Spennarth's process), salts of persulphuric acid and thionyl chloride.

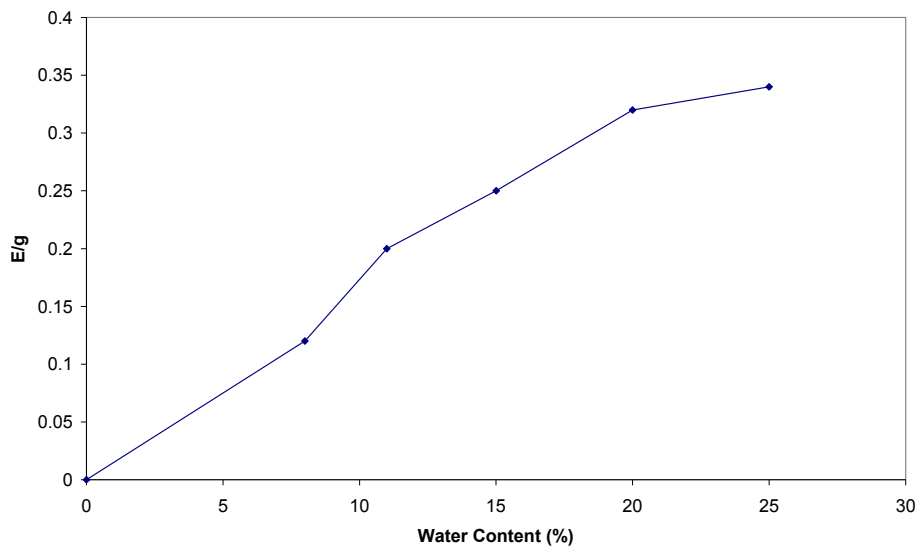
Table 8.1 Effect of acid content on alkali solubility of carbonised wool. Source: Robinson and Westmoreland (1989).

Process	Acid content (%)	Alkali solubility (%)
Uncarbonised	0.17	12.6
Sample A	4.09	16.1
Sample B	5.15	21.0

8.5 Moisture removal

It is essential that as much moisture as possible is removed from the wool prior to drying and baking in order to reduce energy costs and to reduce the risk of damage due to excess acid on the wool fibre. A good squeeze press is necessary, sometimes a double squeeze is used, and in some cases this may be followed by a continuous basket centrifuge. A commonly used example of the latter is sold by Roussellet. Moisture contents of 60% and over have been reported as being detrimental to the process in terms of reduced tensile strength and increased alkali solubility and Wang and Pailthorpe (1989) have demonstrated the relationship, between wool hydrolysis and water retention (Figure 8.1). Wet feed, or acid, hoppers are often used prior to subsequent drying to allow for further adsorption of acid onto the VM and to ensure a continuous, even feed to the dryer.

Figure 8.1 Relationship between moisture content and wool hydrolysis.
Source: Wang and Pailthorpe (1989).



8.6 Drying and baking

This is probably the most critical phase in the carbonising process. To reduce the risk of damage to the wool fibre it should be dried to a low moisture content at a low temperature, preferably 70°C or less. Wang and Pailthorpe (1989) have demonstrated, the effect of drying temperature on wool hydrolysis (Figure 8.2). Ideally, all of the sulphuric acid on the wool should be chemically absorbed which minimises hydrolysis damage to the wool. Once dry, the wool is baked at 95°C to 120°C, where, under the influence of the concentrated acid, the VM is carbonised. Wang and Pailthorpe (1989) have demonstrated the effect of baking temperature on wool hydrolysis, (Figure 8.3). Acid picked up by the VM is only adsorbed onto the surface, so during drying this concentrates on the VM and begins the process of attacking the VM. As the water is evaporated, the acid, which has a great avidity for water, draws the moisture from the VM, which naturally holds the moisture longer. In doing so the VM is converted into carbon compounds that crumble readily. The use of salts of persulphuric acid in the acidising bowl has been suggested, since these accelerate the degradation of the lignin and so can reduce baking times. It is not known if these have been tried successfully on a commercial basis though. High acid concentrations on the fibre, particularly localized concentrations due to uneven wetting, can lead to chemical modification of the wool fibre during baking. This can result in uneven or skittery dyeings due to dye resist effects.

Figure 8.2 Relationship between drying temperature and wool hydrolysis.
Source: Wang and Pailthorpe (1989).

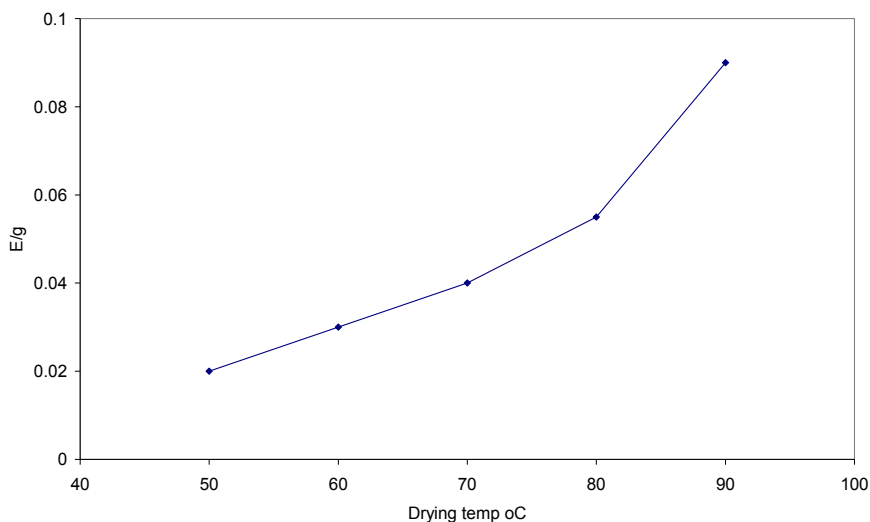
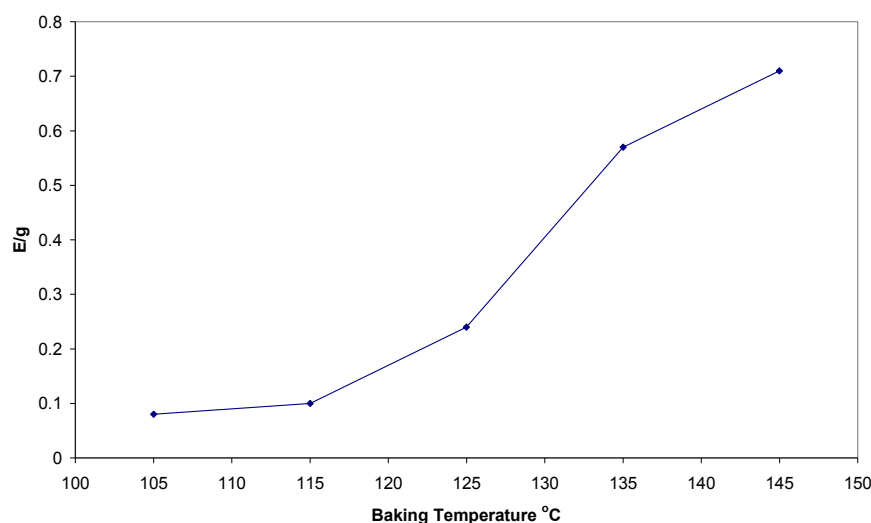


Figure 8.3 Relationship between wool hydrolysis and baking temperature.
Source: Wang and Pailthorpe (1989).



8.7 Burr crushing and dedusting

After the baking stage the treated wool is conveyed to the crushing and de-dusting stages. Depending on the product being processed these stages can be repeated two or even three times to ensure complete removal of the charred VM. In the crushing procedure the wool passes between a series of heavy, fluted, crush rollers that pulverise the carbonised matter. These rollers are often set up such that the speed of the rollers increases slightly from entry to exit zone and between top and bottom. This 'drafting' appears to improve the efficiency of the crushing action. Some work has been done on burr crushing, in particular the damage that can be caused when fibres become trapped in the fluted rollers and are rubbed because of the differential speeds of the top and bottom rollers. Effective crushing with minimum damage was found to be possible when both top and bottom sets of rollers were run at the same speed if a sufficiently thin layer of wool was processed. De-dusting of the crushed VM is accomplished in either a rotating helical drum type duster, or willey, or in a stepped inclined cleaner. It is important that the crushing and dedusting stages are properly set up and monitored if considerable losses in mass are to be avoided (Townend & Russell 1980).

8.8 Neutralisation

In order to remove the excess acid and to make the wool suitable for subsequent processing, neutralisation is required. The principle being based on the salt forming properties of the acid when brought into contact with the alkali. The most commonly used alkali is sodium carbonate. Neutralising is sometimes preceded by a water only rinse in an attempt to reduce chemical usage and minimise any chemical damage that may occur due to the heat of reaction between sulphuric acid and sodium carbonate. In the neutralising bowl the carbonate is converted to bicarbonate and then to the sulphate and free carbonic acid. In order to maintain the finished wool at a pH of between 6 and 8 it is important to maintain the correct balance of carbonate and bicarbonate in the bowl. The neutralising process also helps to remove any residual dust remaining after the de-dusting operations.

A good deal of work has been done on the neutralisation stage of the carbonising process. Although most work has centred around soda ash, the most commonly used of the alkaline substances, there has been some interest in the use of alternatives to soda such as ammonia, ammonia and ammonium acetate and ammonium carbonate, but these have often been found to produce a slight dulling in the appearance of the wool. High acid contents and high baking

temperature have been found to result in significant losses in mass during neutralising due to solubilisation of hydrolysed protein. Prior to drying, the neutralising stage is often completed through a standing bowl containing hydrogen peroxide, usually at a concentration of 1 volume, adjusted to about pH5 with formic acid. The carbonising process is often carried out on the poorer quality wools, which can be quite yellow in appearance, and with better wools can impart some greying or yellowing to the wool. Steeping in hydrogen peroxide can reverse this giving the wool a brighter appearance.

8.9 Machinery

Companies including Fleissner, Annett and Darling, Petrie and McNaught, Dyson, Ardara and Charpentier have all been active in the carbonising field. Until recently Fleissner still produced their carbonising line that was based on the floating wool/suction drum principle and was claimed to give better adsorption of the acid. Fleissner also recommended drying at 60°C in their suction drum dryer. Annett and Darling installed four 2m wide carbonising lines in Australia in the early 90s, at Elders, Riverina and Michells, which utilised the multi-hopper bottomed style of bowl and drum dryers, of these only the two lines at Michell are still operating. The Elders line, having since changed hands through Conagra and Melbourne Scouring Co. has been sold into China. The Riverina line was converted into conventional scour lines. A second hand 1m wide Petrie and McNaught conventional style line has been installed in South Africa. The most recent installation has been at Victoria Wool Processors where the new line, installed in the mid 90's, was a combination of Andar and Ardara equipment. This line has the flexibility of being operated as a single carbonising/neutralising line or as two scouring lines. A bypass conveyor was installed to direct the scoured wool around the acid bowl.

8.10 Testing and control

There are a number of variables within the carbonising operation that should be carefully controlled. These are acidification time, acid concentration of the bath, squeeze roller pressure, hydro-extraction time and G-force, time and temperature in drying and baking zones, crushing and deduster settings, pH of neutralising bowl and carbonate/bicarbonate concentrations, bleaching bowl concentration and pH and temperature of final drying stage. Parameters measurable on the wool are residual wax after scouring, acid content, water content, alkali solubility and pH. The most commonly used test for chemical fibre damage is the alkali solubility test. Unfortunately the test is slow so cannot be used as an on-line QC tool and few mills, if any, are set up to carry out this test. The service is usually provided by independent test houses. A normal scoured wool would have an alkali solubility of around 10-12% whereas a carbonised wool can be anywhere in the range 15-20%. In extreme cases, where carbonising has been poorly controlled and excessive bleaching has been performed, alkali solubilities of up to 30% have been noted. Another test, used solely in research laboratories, is the determination of N-terminal amino acids (WSR 1971) which gives a measure of the hydrolysis. At the mill, testing is usually limited to measurement of the acidising bowl concentration and pH of the neutralised wool.

8.11 Types of wool carbonised

In the woollen industry the decision to carbonise or not depends on the quantity of VM present, the type of VM present and the final product into which the wool is destined. In Australia wool with between 2.1% and 7% VM is classified as Light Carbonising and above 7% as Carbonising types. Around 10% of the clip is so classified. In practice blends with in excess of 1.5% VM are often carbonised either as the loose stock or in the piece if a totally fault (VM) free product is required.

The decision on whether or not to carbonise is most influenced by the type of VM present in the wool. Certain types of VM are easily removed in carding, for example Bathurst Burr, whereas others, such as seed and shive, are removed with difficulty and can cause serious cloth faults if not removed. But there comes a point when the amount of "easy to remove" VM becomes too high and attempts to remove it mechanically in carding can result in serious fibre loss and breakage. So, in general and notwithstanding the comment above, wools with less than 7% VM as burr may not be carbonised whilst on the other hand wools with more than 2% VM as seed and shive will more often than not be carbonised.

8.12 Conclusions

In conclusion it can be said that as a result of the research that has been carried out it has emerged that the Sulphuric acid process is the most viable at present. Also, undue damage to the wool fibre can be avoided by careful monitoring of the carbonising process. Parameters that should be monitored are:

- acid content
- moisture content of the wool entering the dryer
- residence time and temperature of baking
- settings of the burr crushing rollers and the deduster
- neutralizing sequence
- residual acid content, pH, alkali solubility, colour and tensile strength of the carbonised product.

Despite the vast amount of knowledge available on the carbonising process, there is still a need for further information about the best conditions for acidising, drying and baking. The same may be said about the effect of various scouring conditions on the carbonising process and the effect of carbonising conditions on different types of wool and subsequent processes.

Readings

The following readings are available on CD:

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5. Wool Science Review, 1965, Carbonising: Recent research in Australia, *Wool Science Review*, vol. 26, International Wool Secretariat, London, p. 25.

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Summary



Summary Slides are available on CD

Carbonising is the chemical process employed for the removal of burrs, shive and other vegetable matter from scoured wool. The technique consists of treating the wool with dilute sulphuric acid and baking the acid treated wool to char the VM so that it can be removed by mechanical means. Unfortunately, carbonising tends to weaken and discolour the fibre and causes losses in weight during subsequent processing, reduces wear resistance of the final product and can adversely affect dye receptivity. Careful control of the operation is required if these adverse effects are to be minimised.

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Glossary of terms

Acidising	The process of immersing scoured wool in a dilute solution of acid, usually sulphuric acid
Baking	The high temperature process where the VM is dehydrated and converted to hydrocellulose (charcoal) after drying
Carbonising	This is the name of the chemical process used to remove vegetable matter from wool
Crushing	The process, usually through a series of fluted steel rollers, for breaking up the charred VM
Dedusting	The process of removing the broken charred VM from the wool. Normally accomplished through a helical willey or step cleaner
Drying	The acidic wool is dried to as low a regain as possible without inducing localised acid damage
Hydrolysis	This is a measure of the damage caused to the fibre by the acid. It is expressed as E/g of wool where E is the optical density of the ninhydrin reaction product per gram of wool. An E/g of 1.0 represents 1.5×10^{-5} mol of NH_2
Neutralisation	After crushing and dedusting the acidic wool is rinsed, soaked in alkali and rinsed again to bring the wool back to a neutral pH
Vegetable matter or VM	This consists of seeds, burrs, grasses, twigs etc

