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Scouring Conditions
Part II

The Effect of Rinsing at Different Liquor pH Values during Scouring on the Subsequent Combing Performance of Wool

by

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THE EFFECT OF RINSING AT DIFFERENT LIQUOR pH VALUES DURING SCOURING ON THE SUBSEQUENT COMBING PERFORMANCE OF WOOL

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ABSTRACT

When wool is rinsed in liquors of different pH values following conventional scouring changes in the entanglement of the wool are introduced which in turn affect combing performance. Combing performance depends on the pH of the liquor in which the wool is rinsed, the reagent used to control pH and the residual grease level of the scoured wool. Under certain conditions such aqueous treatment may improve combing performance.

KEY WORDS

Wool – pH of liquor – pH of scoured wool – alkaline scoured – neutral scoured – residual grease – entanglement – combing performance.

INTRODUCTION

This paper is the second of a series in which the objective is to arrive at a set of scouring conditions which will give optimum results in subsequent mechanical processing.

In part I of this series of publications the combing performance of backwashed slivers prepared from neutral scoured wool was investigated[1]. The pH of the backwash liquor was changed by using different reagents at different concentrations. In addition, wools of different style were used. The combing performance of these slivers was found to depend on the pH of the liquor and on the reagent used to control the pH. The performance deteriorated with deterioration in wool style and this was accentuated at liquor pH values of above 10,5. Changes in performance within a particular style were possibly brought about by changes in entanglement due to differential relaxation of the fibres in the different media. Certain maxima and minima were observed in these curves some of which may have been related to the point of zero combination and also the iso-electric point. It was not clear, however, whether some of the patterns obtained in the aforementioned work would be applicable to raw wool scouring since the wool would be in a loose, instead of sliver, form and furthermore a carding operation would be introduced

between scouring and preparing. It was therefore decided to conduct similar experiments as previously but to use raw wool as the starting material and to introduce different reagents at different pH values into the *rinse bowl* of the *scouring train.* Scouring procedures were both alkaline and neutral.

EXPERIMENTAL

Raw materials

Four different lots of grease wool of approximately 64's quality and good topmaking style were used in the experiments described, a different lot being used for each series of experiments. Three lots were of 9/11-months length and one lot was of 12-months length.

Scouring

The wool was scoured in a four bowl Petrie & McNaught pilot scale scouring plant equipped with a Fleissner suction drum drier. Since the scouring plant comprised only four bowls utilisation of the equipment varied between series of experiments as set out below.

1st Series: (Alkaline scour)

A constant amount of soda ash (1 g/litre) was used in the first bowl. Varying amounts of detergent were used in the first and second bowls to produce variations in residual grease. A small amount (0,03 g/litre) of detergent was used in the third bowl. Either formic acid or sodium carbonate was used in both the third bowl and the fourth bowl (rinse bowl) at the desired pH level. Temperatures of the liquor in the four bowls were 55°, 55°, 50° and 40°C respectively.

2nd Series: (Alkaline scour)

The grease wool was scoured under commercial conditions with soda ash and detergent and then dried and blended and re-introduced into the scouring train. At this stage the residual grease was 0,5 *per cent* and the pH of the aqueous extract of the wool was 9,2. On this second passage through the scouring train no detergent was used and all the bowls were charged with either formic acid or hydrochloric acid at the desired pH level. The temperatures of the liquor in the bowls were 18°, 18°, 18° and 40°C respectively during the second passage.

3rd Series: (Neutral scour)

The grease wool was scoured in four bowls to a residual grease level of 1,1 *per cent* using a non-ionic detergent only and then dried and blended and re-introduced into the scouring train. During the second passage the first bowl contained tap water only and the following three bowls were charged with sodium carbonate (in one experiment a mixture of equal proportions by mass of sodium carbonate and sodium hydroxide was used) at the desired pH level. The temperatures of the liquor in the bowls were 45°, 45°, 40° and 40°C respectively during the second passage.

4th Series: (Neutral scour)

The grease wool was partially scoured in four bowls to a residual grease level of 3,0 *per cent* using a non-ionic detergent only and then dried and blended and re-introduced into the scouring train. On the second passage various concentrations of a non-ionic detergent was used in the first bowl in order to produce variations in residual grease. The remaining bowls were charged with either formic acid, sodium carbonate or sodium hydroxide at the desired pH level. The temperatures of the liquor in the bowls were 55°, 45°, 45° and 40°C during the second passage.

In all experiments the wools were dried at 60°C and a carding lubricant was applied as the wools emerged from the drier. The amount applied was the same in all cases and effected an increase in the ether extractible matter level of the scoured wool of 0,3 *per cent.*

In some experiments of the fourth series the wool was rinsed in tap water only and was sprayed with a solution of formic acid or sodium carbonate together with the carding lubricant *after drying* and in one experiment the wool was sprayed with formic acid *without* the carding lubricant.

During these trials great care was taken to ensure that all settings, production rates etc. were maintained constant within each series. The settings used and procedures followed in subsequent mechanical processing were also maintained constant.

Subsequent mechanical processing

After conditioning for one hour following scouring each lot was carded, gilled three times and combed. These processes were executed without delay and in all cases were carried out at 70% relative humidity and at a temperature of 21°C.

The wool was carded on a double swift F.O.R. Continental worsted card the swifts and doffers of which were clothed with metallic wire. The regain before carding was 15 *per cent* which was near to the optimum value required for a subsequent procedure which included the application of water during gilling[2]. Gilling was carried out on an NSC gillbox with fallers of pin density 6,5 p.p.cm. Water was applied to the ingoing slivers in both the first and second gillings to increase the regain to that required for optimum combing performance[2]. The material was combed on an NSC PB26 comb with the gauge set at 26. Four tests were carried out at 15 minute intervals to determine the percentage noil.

Testing

The pH values of the scouring liquors were monitored using a Metrohm pH meter and readings checked from time to time. Values published in this paper are those obtained after cooling samples taken from the liquor to 20°C. The pH of the scoured wool was measured according to the I.W.T.O. method. Total alkali was measured according to the method of Veldsman[3] and total acid measured according to the I.W.T.O. method. Residual grease was measured by the rapid column and tray method using ether as solvent. Regain was measured by means of the direct reading CSIRO regain tester.

RESULTS AND DISCUSSION

1. Rinsing of an alkaline scoured wool at various residual grease levels in liquors of different pH values

Figure 1 shows the percentage noil obtained during the combing of the wool used in the experiment and which had been rinsed at liquor pH values of 2,8 and 10,8. (pH values of the aqueous extracts of the scoured wools produced were 3,1 and 9,4 respectively).

It is clear from Fig. 1 that rinsing of the wool in aqueous media at pH 2,8 resulted in a significant improvement in combing performance compared with rinsing in aqueous media at pH 10,8. In the case of the wool lots which were rinsed at pH 2,8 optimum performance was achieved when the residual grease level was approximately 0,4 *per cent*. The percentage noil was extremely low in all experiments, even for a wool of spinners style, and indicated that processing must have taken place under nearly ideal conditions. Nevertheless the relative improvement in percentage noil between that obtained for a liquor pH of 10,8 and that obtained for a liquor pH of 2,8 at optimum ether extractible matter was of the order of eight *per cent* (0,2% absolute).

2. Rinsing of an alkaline scoured wool having constant residual grease in liquors of different pH values

Since residual grease was constant (0,5%) for all the wool lots used in this experiment it was possible to determine the relationship between percentage noil during combing and the pH of the liquor in which the wool had been rinsed for this particular level of residual grease. This is illustrated in Fig. 2.

From Fig. 2 it can be seen that the curve obtained for formic acid shows a maximum at a liquor pH in the vicinity of 6,5 and a minimum at a liquor pH in the vicinity of 4. In the former case this pH corresponds with that reported previously[1] and appears to be related to the point of zero combination. In the latter case the pH is lower than that reported previously for formic acid. The previous report, however, dealt with *neutral* scoured wool whereas in this experiment an *alkaline* scoured wool was used. Furthermore, the immersion times and temperatures were different. A lower pH of the liquor in the rinsing bowls may therefore have been necessary to produce the iso-electric effect. This is supported by the data given in Table 1 which show that only liquor pH values of below 4,5 produced an acid reaction in the test for acid content on the resultant scoured wool. Furthermore it can be seen that there was no detectable combination of acid with the fibre at a liquor pH of 6,4.

Two points in Fig. 2 represent the results obtained with the use of hydrochloric acid. If these two points are joined with the point representing water a curve such as that shown by the broken line can be obtained. The curve drawn is

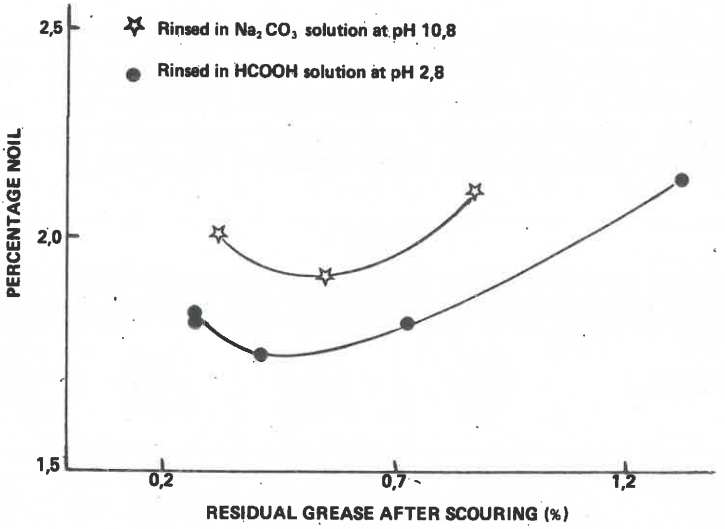

FIGURE 1

Percentage noil obtained for various residual grease levels after rinsing of an alkaline scoured wool in sodium carbonate and formic acid solutions

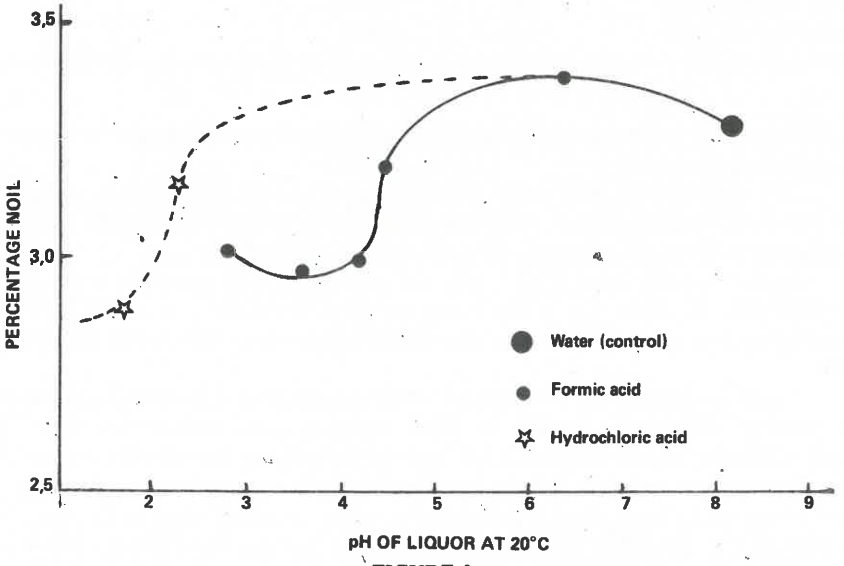

FIGURE 2

Percentage noil obtained for a constant residual grease level of 0,5% after rinsing of an alkaline scoured wool in formic acid and hydrochloric acid solutions

based upon curves previously obtained by the author since it satisfies the results previously reported upon in so far as the maximum is common to both acids and the minimum shifts to lower pH values with increasing acid strength.

From the figure it is clear that combing performance could be improved by aqueous rinsing in acid media under certain conditions of liquor pH.

TABLE 1

ACID AND ALKALI CONTENTS OF ALKALI SCOURED WOOL AFTER RINSING IN FORMIC ACID AND HYDROCHLORIC ACID SOLUTIONS

Reagent	pH of rinsing liquor	Acid content (m.eq/g)	Alkali content (m.eq/g)	pH of wool
Water (control)	8,2		0,036	8,3
Formic acid	6,4		0,037	8,3
Formic acid	4,5		0,023	7,8
Formic acid	3,6	0,019		4,9
Formic acid	2,8	0,115		3,3
Hydrochloric acid	2,3	0,063		3,5
Hydrochloric acid	1,7	0,207		2,9

3. **Rinsing of a neutral scoured wool having constant residual grease in liquors of different pH values**

The wool used in this series of experiments had a residual grease content of 1,1 *per cent*. Percentage noil results are given in Fig. 3 together with the results for vegetable particles and neps present in the slivers after carding.

It can be seen from Fig. 3 that the percentage noil showed a tendency to increase with increasing pH of the liquor except in the vicinity of pH 10,5 at which a minimum was apparent. In a previous study minima were also found at this liquor pH[1].

Samples taken from the scoured wools produced at the various pH levels of the rinsing liquor were analysed for alkali solubility and lanthionine content. The alkali solubility varied between 15,4 and 17,4% and the lanthionine content was less than 0,1% in all cases. Bundle breaking strength tests were carried out on the card slivers made from these wools and the values obtained were in the range 10,5 to 10,8 gf/tex. From the above results it seemed that any fibre damage which may have taken place was difficult to detect and therefore probably of little significance. The differences observed in combing performance, however, suggest that some mechanism was involved during pH alteration of the wool.

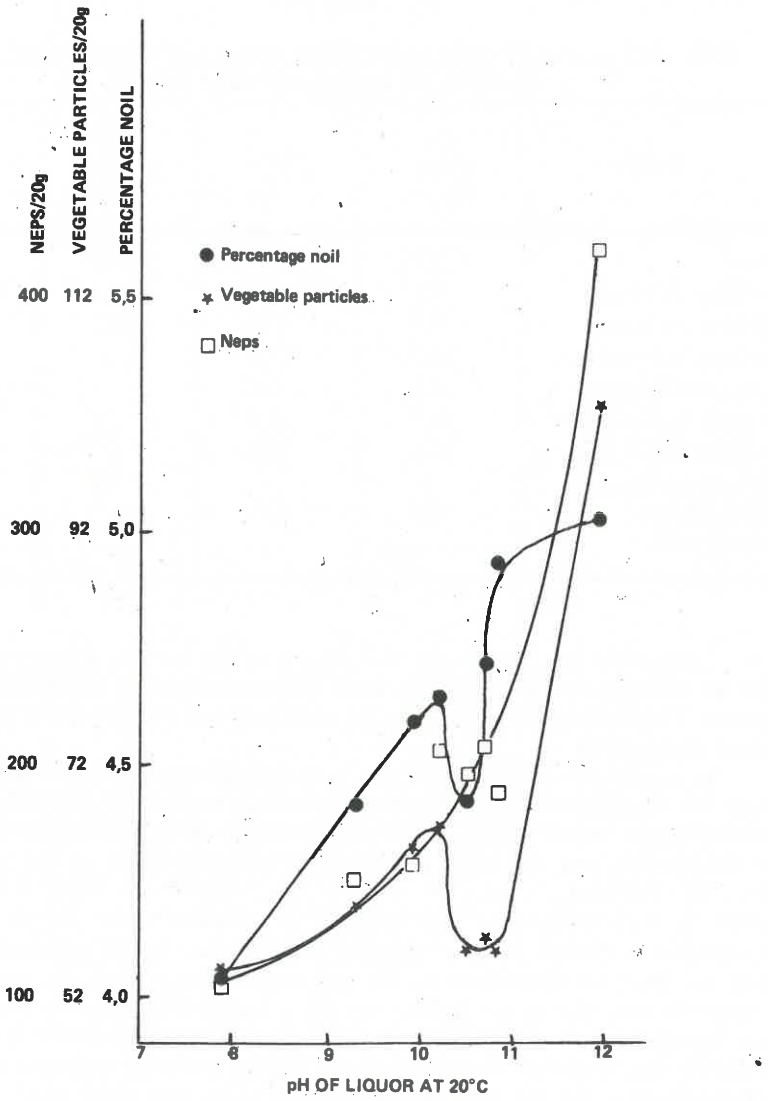

FIGURE 3

Neps and vegetable impurities after carding and percentage noil after combing for neutral scoured wool having constant residual grease of 1,1% rinsed in alkaline media

TABLE 2

ACID AND ALKALI CONTENTS OF NEUTRAL SCOURED WOOL AFTER RINSING IN ALKALINE SOLUTIONS

Reagent	pH of rinsing liquor	pH of wool	Acid content (m.eq/g)	Alkali content (m.eq/g)
Water	7,9	6,9	0,013	
Sodium carbonate	9,3	6,9	0,013	
Sodium carbonate	9,9	6,9	0,012	
Sodium carbonate	10,2	7,5		0,030
Sodium carbonate	10,5	8,5		0,044
Sodium carbonate	10,7	8,9		0,053
Sodium carbonate	10,8	9,3		0,069
Sodium carbonate plus Sodium hydroxide	11,9	10,5		0,146

From table 2 it can be seen that rinsing of the wool in liquors up to pH 9,9 had no effect upon the pH of the wool and no noticeable effect upon the acid content. Combination of alkali with the wool was only clearly evident at liquor pH values of above 9,9.

In a previous publication[1] the author suggested that under certain conditions the vegetable count of slivers subjected to mechanical treatment could be related to the initial entanglement of the fibres. It is interesting to note that in the present case the curve for vegetable particles shown in Fig. 3 resembles that for percentage noil, so that if the concept of using the vegetable count as an indication of entanglement is accepted under certain controlled conditions a clearer picture emerges. Differences in entanglement may or may not produce differences in the nep count after carding but again it is of interest to note that in the present case the correlation between the nep count and the vegetable count was found to be highly significant (0,1% level). It would seem therefore that variation in the pH of the liquor produced variations in entanglement probably by the same mechanism as previously reported[1] namely a differential relaxation of the fibres in the different media. These variations in entanglement resulted in variations in the number of vegetable particles held firmly by the fibres during carding and also in variations in the neppiness of the card sliver. The higher the entanglement the greater the number of neps which were formed and the greater the number of vegetable particles retained after carding.

TABLE 3

pH OF THE AQUEOUS EXTRACT OF SCOURED WOOLS AFTER RINSING IN LIQUORS OF DIFFERENT pH VALUES AND AFTER SUBSEQUENT SPRAYING

Reagent used in rinsing liquor	pH of rinsing liquor	Reagent sprayed onto wool after drying	pH of aqueous extract of wool
Water	8,0	–	6,7
Water	8,0	Formic acid	4,6
Water	8,0	Sodium Carbonate	7,3
Formic acid	4,5	–	5,2
Formic acid	3,7	–	4,0
Sodium carbonate	9,6	–	7,0
Sodium carbonate	10,4	–	7,6
Sodium carbonate	10,9	–	9,2
Sodium hydroxide	11,3	–	8,7

4. **Rinsing of a neutral scoured wool at various residual grease levels in liquor of different pH values**

Results obtained for percentage noil in this experiment are shown in Figs. 4, 5 and 6. The effect of rinsing at various pH values of the liquor on the pH of the aqueous extract of the wool is shown in Table 3.

From Fig. 4 it will be seen that the relationship between percentage noil and residual grease was practically linear in the case of the control test (which was rinsed in water at pH 8) whereas the relationship showed minima when higher pH values were used. These minima appeared to be more pronounced as the pH increased and furthermore displayed a tendency to occur at higher residual grease levels with increase in pH. The curve shown for the results of the lots rinsed with sodium hydroxide differed from the curves obtained when sodium carbonate was used and it would seem that combing performance was possibly more sensitive to residual grease changes when the former reagent was used. The results depicted in Fig. 4 thus serve to show that the optimum combing performance of wools which have been rinsed in alkaline solutions follows a pattern which is dependent on the pH of the liquor, and the residual grease. In some cases, such as when the residual grease level was about 0,7 *per cent* combing performance tended to improve with increase in the pH of the liquor in the alkaline range. In other cases, such as when the residual grease level was between 0,35 and 0,2 *per cent* combing performance tended to deteriorate with increase in pH of the liquor in the alkaline range.

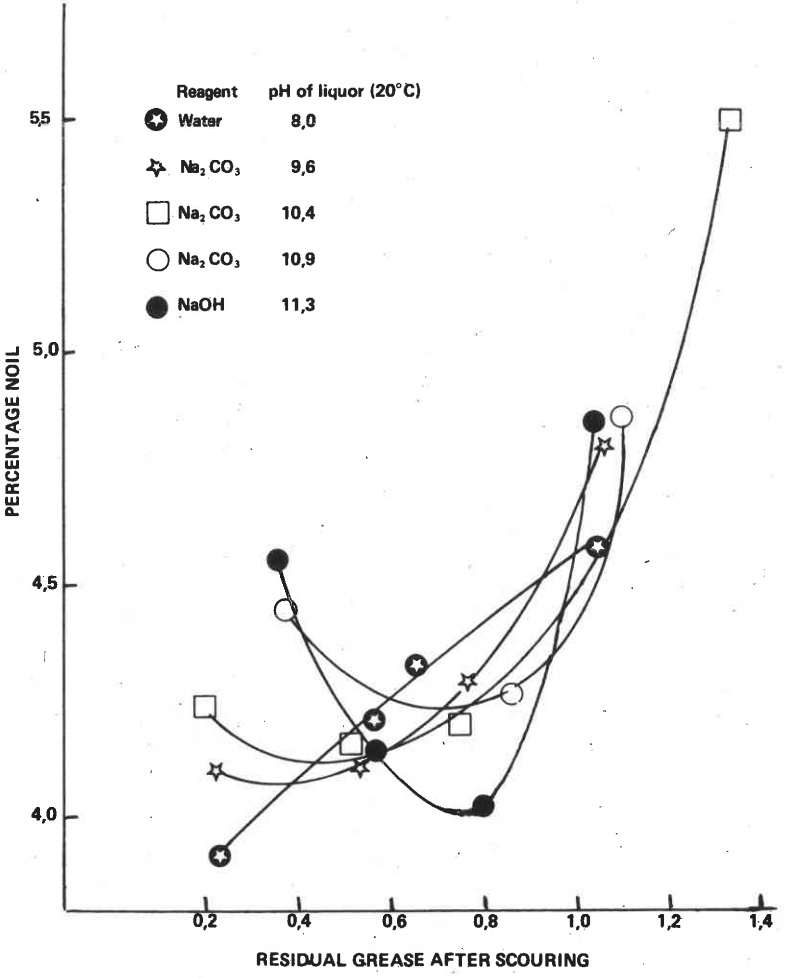

FIGURE 4

Percentage noil obtained for various residual grease levels after rinsing of a neutral scoured wool in sodium carbonate and sodium hydroxide solutions

The results obtained when the wool was rinsed in liquors having pH values of 3,7 and 4,5 are shown in Fig. 5. It appears from this figure that optimum performance tended to occur at residual grease levels which lay in the vicinity of 0,3 *per cent*. When the pH of the liquor was 3,7 combing performance was better than when the pH of the liquor was 4,5 irrespective of the residual grease level. The best performance obtained was, however, not quite as good as the optimum performance recorded for the control.

When the wool was passed through the wash bowls containing water at pH 8,0 (as with the control test) and was subsequently sprayed with solutions of sodium carbonate or formic acid a rather interesting set of results was obtained (see Fig. 6). When a carding lubricant was used (as was the case in all the experiments so far described) the relationship between percentage noil and residual grease was linear. When the carding lubricant was omitted the percentage noil obtained increased rapidly below a residual grease level of approximately 0,7 *per cent* but at and above this value it appeared to be of the same order as that obtained for the same total fatty matter level when the carding lubricant was included. The use of a carding lubricant was thus shown to be essential for the good combing performance of wools containing low to medium residual grease.

In all experiments in this series the number of vegetable particles before combing lay in the range 43 to 52 particles per 20 grams, emphasising the extremely clean nature of the wool selected for the experiment. Under these circumstances and more particularly since residual grease was a variable it was not possible to expect nor to find a relationship between the vegetable count and either the combing performance or the nep count. On the other hand the number of neps in the carded slivers was of a much higher order and ranged from 150 to 300 neps/20 g. A statistical analysis of the results for percentage noil and neps showed that the correlation between these two parameters was highly significant (0,1% level).

It is reasonable to suppose from the evidence built up so far during the work on aqueous pH treatment that changes in entanglement take place when wool is subjected to rinsing in aqueous media at different pH values. This can be reflected by either the vegetable count or the nep count (or sometimes both), of carded slivers from that wool, depending upon the circumstances of the case.

SUMMARY AND CONCLUSIONS

Various studies were made to establish the effect on subsequent combing performance of an aqueous rinse at different pH values on wools which had previously been scoured in either alkaline or neutral media. In some cases the residual grease content was constant and in other cases it was varied.

It was shown that when wools had previously been scoured in an alkaline medium subsequent rinsing in acid medium could improve combing performance. Optimum performance was dependent on the residual grease level and the pH of the liquor.

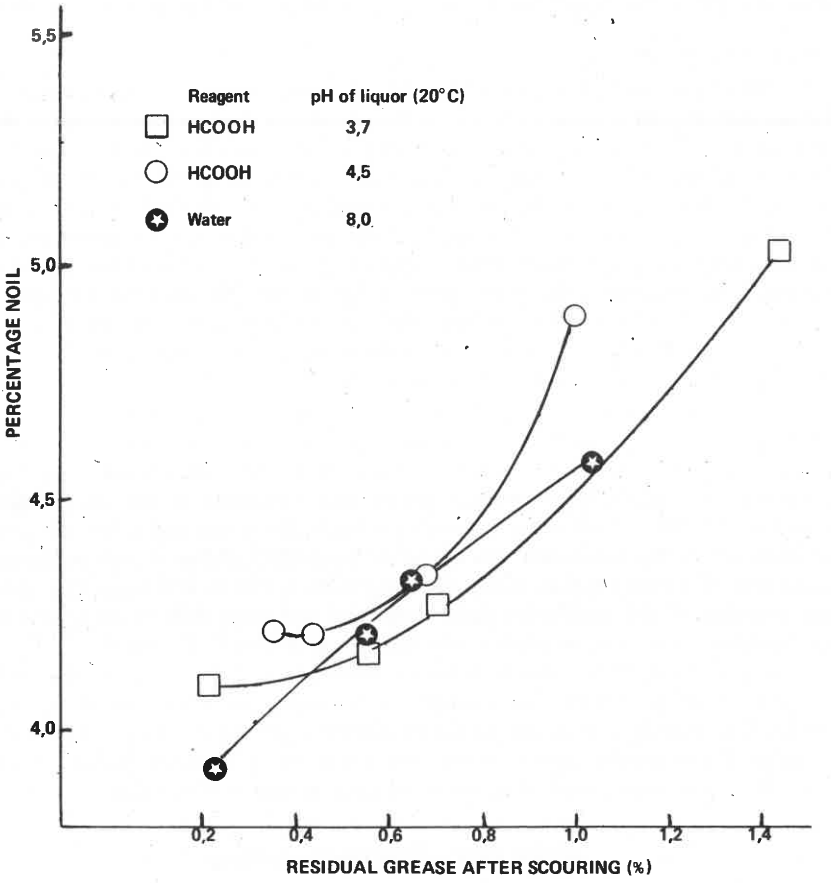

FIGURE 5

Percentage noil obtained for various residual grease levels after rinsing of a neutral scoured wool in formic acid solution

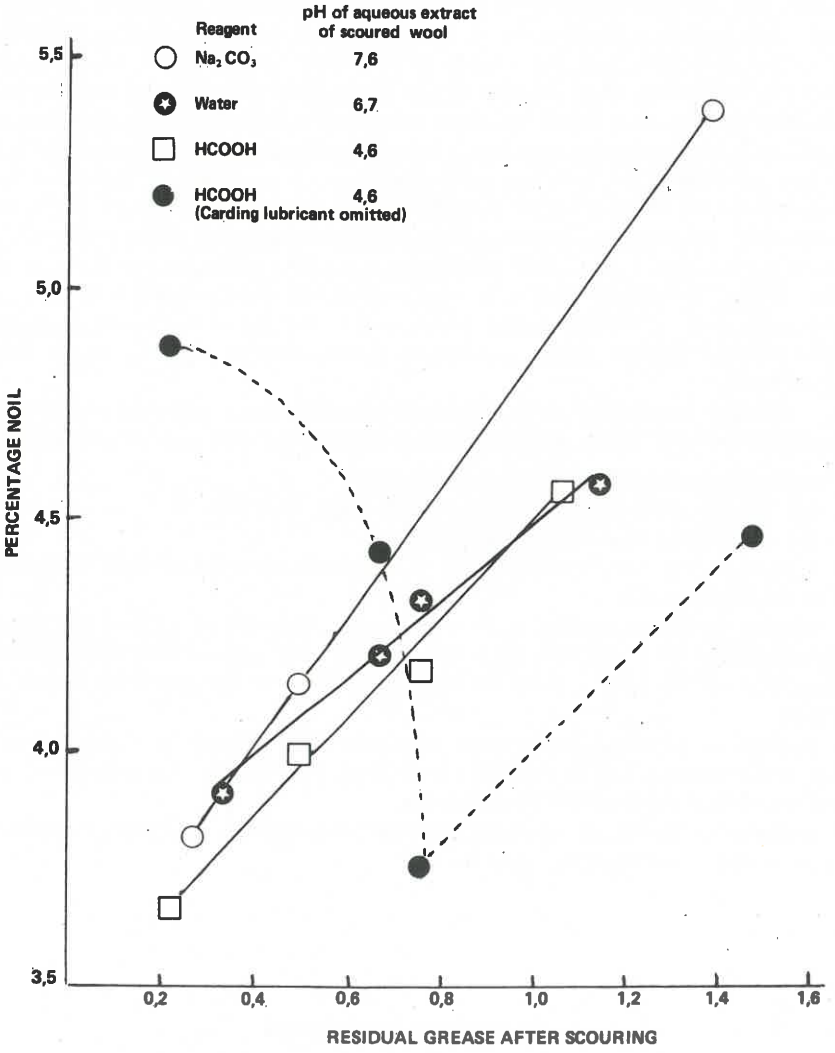

FIGURE 6

Percentage noil obtained for various residual grease levels after spraying wool previously rinsed in water with sodium carbonate and formic acid solutions after drying

For wools which had previously been scoured in a neutral medium it was shown that the advantages of rinsing in liquors at different pH values were dependent on the residual grease level. It was possible to improve combing performance by using higher pH values of the liquor provided the residual grease was of the order of 0,7% but at low (0,2) residual grease levels such treatment resulted in poorer combing performance. When the wool was rinsed in aqueous media without acid or alkali a linear relationship was found between percentage noil and residual grease. At low residual grease combing performance of wool rinsed in water was better than either that of wool rinsed in alkaline or acid solutions although only two acid values were investigated. The linear relationship obtained for wools which had been rinsed in water held even after subsequent spraying of the wool with acid or alkali after drying. Rinsing in liquors in which alkali was used produced minima in the percentage noil – residual grease curves and as the pH of the liquor increased so these minima became more pronounced and tended to occur at higher residual grease levels.

Certain similarities were observed between these experiments and those described in Part 1 of this series of publications insofar as it was observed that

1. combing performance was dependent upon
   (a) the pH of the liquor in which the wool was treated;
   (b) the reagent used for controlling pH.
2. a maximum in the percentage noil – pH curves occurred at a pH of the liquor of approximately 6,5;
3. minima in the percentage noil – pH curves occurred at a pH of the liquor of approximately 10,5 and also at a value in the acid region. This latter value was dependent on the reagent used but may also have been dependent on certain other factors;
4. changes in combing performance were related to changes in entanglement and that such changes could be reflected by changes in either the vegetable count or the nep count (sometimes both) of the slivers;
5. combing performance could be improved by aqueous treatment at certain pH values under certain specific conditions.

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