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Scouring Conditions Part III: A Preliminary Study on the Effect of pH and Temperature during Grease Wool Scouring on Neps and Percentage Noil

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SCOURING CONDITIONS

PART III:

A PRELIMINARY STUDY ON THE EFFECT OF pH AND TEMPERATURE DURING GREASE WOOL SCOURING ON NEPS AND PERCENTAGE NOIL

by D. W. F. TURPIE

ABSTRACT

A preliminary study was made to determine the effect of liquor pH (using soda ash) and temperature on neps and percentage noil during conventional grease wool scouring to a residual grease level of approximately 0,4 per cent. The amounts of wool used were small and no repeat experiments were undertaken. Unavoidable differences in scouring efficiencies placed certain limitations on the conclusions.

If soda ash was used in the first bowl only, results favoured a pH of the liquor preferably not exceeding 9,5 and a temperature below 60°C. If the resulting pH values of the aqueous extract of the scoured wool were too low, however, the results favoured pH adjustment of the liquor in the second bowl, and this was also considerably more economical. If no soda ash was used the results favoured a liquor temperature of 60/65°C.

KEY WORDS

Grease wool – aqueous scouring – pH of liquor – temperature of liquor – soda ash – detergent – neps – percentage noil.

INTRODUCTION

This paper is the third of a series[1, 2] in which the objective is to arrive at a set of scouring conditions which will affect subsequent mechanical processing most favourably.

In part II of this series[2] different reagents, at different pH values, were introduced into the *rinse bowl* of the scouring train after conventional scouring in both alkaline and neutral media. It was found that this procedure resulted in changes in the entanglement of the wool which, in turn, affected combing performance. Combing performance was found to depend on the pH of the liquor in which the wool had been rinsed, the reagent used to control pH and the residual grease level of the scoured wool. Under certain conditions such aqueous treatment was found to result in improved combing performance; under others, combing performance deteriorated. When the residual grease level was around 0,35 *per cent,*

for example, combing performance tended to deteriorate slightly with an increase in the pH of the liquor from 8,0 to 10,4 and to deteriorate very rapidly at a pH value of 10,9 using soda ash as reagent. The nep count of the carded slivers in some of the experiments described was correlated with the results for percentage noil at the 0,1% level of confidence.

It was previously reported that there are certain limits of pH within which successful *scouring* can be carried out[3]. The upper limit for efficient scouring is the highest pH value at which the wool can be scoured without danger of damage to the fibre. High concentrations of soda ash or detergent during conventional aqueous scouring should be avoided since both conditions are liable to result in felting of the fibres, particularly at elevated temperatures, and are, in addition, uneconomical. It was suggested that the pH of the liquor in the first bowl could safely rise to 10 or just over, but that in the second bowl it was not advisable to exceed the value of 10 to any marked extent[3]. It was still not clear, however, as to what the effect of pH of the liquor during the *initial* stages of scouring was on subsequent *processing performance.*

Since so many parameters had already been shown to have an effect upon the performance when the pH was changed during rinsing, it was decided in the present investigation to attempt to keep the residual grease level of the scoured wool constant at a popular commercial level of about 0,4 *per cent.* It was also decided to study the effect of variations in pH (using soda ash) and in temperature of the liquor in the first and second scouring bowls on neps and percentage noil. This could, therefore, be regarded as a preliminary study as further investigations using other reagents and other residual grease levels would be necessary to complete the picture. It was possible to carry out only a limited number of small experiments within the available budget.

EXPERIMENTAL

(a) **Raw Materials:**

A nine-bale lot of 9/11 months fleece wool of mean fibre diameter 21,1 μm and having a crimp frequency of 5,4 crimps per cm was selected for this investigation. The yield of this wool was estimated to be approximately 55 *per cent* on a drycombed top and noil basis. The wool was carefully layer-blended and vertical slices, each of a mass of 40 kg, were taken from the blend for each experiment. Altogether twenty experiments were undertaken.

(b) **Scouring:**

The wool was scoured in a one-foot wide four-bowl Petrie & McNaught pilot scale scouring plant equipped with an automatic feed and a suction drum drier. A side settling tank was used in conjunction with both the first and second bowls so that the total capacity of each of these bowls was 1 300 litres. The capacities of the third and fourth bowls were 455 litres each. All squeeze rollers (each 300 mm wide)

were set at a pressure of 3 000 kg. The speeds of all squeeze rollers and rakes were 6 r.p.m. and 11 cycles per minute, respectively. The temperature of the drier was thermostatically controlled at 70°C. The greasy wool was fed to the scouring train at a controlled rate of 100 kg per hour. During each experiment the feed was interrupted four times at specific intervals to await the results of grease tests and to make appropriate adjustments to the rates of addition of detergent.

Two series of experiments were carried out. In the first series a set of four experiments was carried out during which *no* pH adjustment was made to the liquor in the first bowl, and a further three sets of four experiments during which this pH was adjusted and maintained at 9,5 10,5 and 11,0 respectively, making 16 experiments in all. The four experiments in each set were carried out at different temperatures of the liquor in the first bowl, namely at 52°, 55°, 60° and 65°C respectively. The temperatures of the liquors in the second, third and fourth bowls were 55°, 50° and 40°C respectively in all experiments (excepting those in which the temperature of the liquor in the *first* bowl was 52°C in which case the temperature of the *second* bowl was also 52°C). In the second series three experiments were carried out during which the temperatures of the liquors in the four bowls were 55°, 55°, 50° and 40°C respectively, and the pH of the liquor in the first bowl was adjusted to and maintained at 9,5. These three experiments were carried out at different specific pH values of the liquor in the *second* bowl, namely 9,5 10,5 and 11,0. A set of four results was obtained by using the above results together with the result pertaining to the first series in which tap water (pH 8,5) was used in the second bowl, the pH of the liquor in the first bowl was 9,5 and the temperatures of the liquors in the four bowls were 55°, 55°, 50° and 40°C respectively.

The bowls were thoroughly cleaned and made up with fresh liquor in the case of every experiment. The pH of the liquor in the first bowl, in the case of the first series of experiments, and in both the first and second bowl in the second series of experiments was then adjusted with the requisite amount of soda ash to obtain the required specific value. In both series the first, second and third bowls were charged with an amount of detergent considered appropriate for the scouring conditions prevailing for the production of a residual grease level on the scoured wool of around 0,4 *per cent*. Further additions of soda ash were made to the liquor in the side settling tank of the relevant bowl(s) at frequent intervals to maintain the pH constant while scouring was in progress. Detergent additions were made four times i.e. once after each interruption of the scouring period, in an attempt to obtain an overall average residual grease level on the scoured wool of around 0,4 *per cent*. The quantities of detergent selected for addition to the various bowls were such that there was a progressive removal of grease and impurity from the wool without any obvious over-scouring or under-scouring at any stage. The purpose of following the above procedures was to try and obtain similar scouring efficiencies in all bowls. Under such ideal circumstances the real effect of pH and temperature of the liquor on the nep count could be observed. The amounts of soda ash and detergent used

were not considered commercial because of the short duration of the experiments.

The fourth bowl was used as a rinse bowl, fresh water being introduced at 400 *l*/hr during the scouring period and displacing an equivalent amount to waste. No backflow was used.

(c) **Carding and combing:**

Each lot of scoured wool was sprayed with 0,2 *per cent* by mass of Oxitex 40 (Shell Chemicals) together with 2,0 *per cent* by mass of water, and then allowed to condition at a relative humidity of 70 *per cent* and a temperature of 21° C for approximately 20 hours before carding.

The wool was carded on a F.O.R. double swift Continental worsted card, the swifts and doffers of which were clothed with metallic wire. The workers were set progressively closer to the swift (from 18 B.W.G. in the case of the first worker to 28 B.W.G. in the case of the last worker). The card production rate was 15 kg/hr at a swift speed of 78 r.p.m.

Three gilling operations on a N.S.C. intersecting gill box followed carding. During the first operation the slivers were sprayed with water to increase the regain for combing to approximately 21 *per cent*. Successive drafts used were five, six and six with the ratch set at 35 mm in each case.

Combing was carried out on a Schlumberger PB 26 comb at a gauge setting of 28 mm. A top comb of pin density 28 p.p.cm and a segment of the Nitto Unicomb type suitable for use with 64's wool were used. Four tests were carried out to determine the percentage noil in each case. The regains of both top and noil were measured in each case so that the percentage noil could be expressed as being under standard conditions.

(d) **Testing:**

The pH values of the scouring liquors were monitored using a Metrohm pH meter. Readings were checked from time to time by measuring the pH on a Metrohm potentiograph, after cooling samples taken from the liquor to 20°C. The residual grease on the wool was measured by the rapid column and tray method using dichloromethane as solvent. Measurement of the pH of the scoured wool was carried out according to the I.W.T.O. method. Regain was measured at all stages prior to combing by means of the direct reading CSIRO regain tester. Regains of the tops and noils were measured by drying to the bone dry state. Mean fibre length measurements on the tops were carried out on an Almeter.

Neps and vegetable particles were counted visually on a Toenniessen top testing machine using transmitted light. Five tests, each involving 200 total faults, were carried out on the card slivers. The same operator counted all the faults. This operator was given a photocopy of four different circles of arbitrarily chosen diameters namely 2,0 1,5 1,0 and 0,5 mm. This was pasted on the ground glass screen of the instrument alongside the path of the fibres, and could be seen through the viewing glass at all times. The operator classified the neps as being $\leqslant$2,0 but $>$1,5, $\leqslant$1,5 but $>$1,0, $\leqslant$1,0 but $>$0,5 and $\leqslant$0,5 mm.

RESULTS AND DISCUSSION

The average trend for the amounts of soda ash which had to be added to the liquor in the *first* scouring bowl to maintain different specific pH values of the liquor in that bowl is shown by a solid line in Fig. 1. The spread of values obtained at the different pH values is also shown in the figure.

It can be seen from Fig. 1 that relatively small amounts of soda ash were required to maintain the pH of the liquor in the first bowl at 9,5 whereas to maintain pH levels of 10,5 and 11,0 required large and very large amounts, respectively. At each pH level the amounts which were used varied as shown by the spread of values in the figure, but these variations did not appear to have been due to temperature. They may have been brought about by variations in other unspecified scouring conditions. The average trend appeared to be exponential with increasing pH.

The broken line in Fig. 1 indicates the total amount of soda ash which had to be added to the liquors of both the *first* and *second* scouring bowls to maintain different specific pH values of the liquor in the *second* scouring bowl while maintaining a constant pH value of 9,5 in the *first* bowl. It is clear that considerably less soda ash had to be added to maintain the pH of the liquor in the second bowl at a value higher than approximately 9,5 than was necessary to maintain the same pH value of the liquor in the *first* bowl. Since both bowls were of the same capacity, it would appear that a significant buffering action in the first bowl must have taken place, probably as a result of the presence of relatively high suint concentrations in the first bowl.

The amounts of detergent used in the experiments to which Fig. 1 relates are given in Tables I and II. The residual grease levels of the scoured wool emerging from the squeeze rollers of the first bowl and after scouring had been completed are also given in the tables. The dichloromethane extractable matter and pH of the aqueous extract after 3 gillings are also given.

From Table I it can be seen that, in general, significantly less detergent was used in the first bowl as the temperature was increased from 52° to 65°C at a specific pH of the liquor. The relevant amounts of detergent used in the second and third bowls also decreased but to a much lesser extent. The total amount of detergent used in all three bowls has been plotted against temperature in Fig. 2, the solid line indicating the average overall trend. It is clear from Table I and Fig. 2 that, in general, as the pH of the liquor in the first bowl was increased less detergent was used at each specific temperature. At 60°C and above, however, the effect of pH appeared negligible and the amounts of detergent used were small.

The purpose of following the above procedures was to try and obtain similar scouring efficiencies in all bowls in every experiment. Due to the small quantities of wool used under such widely differing conditions, however, some relatively wide fluctuations were, understandably, to be expected.

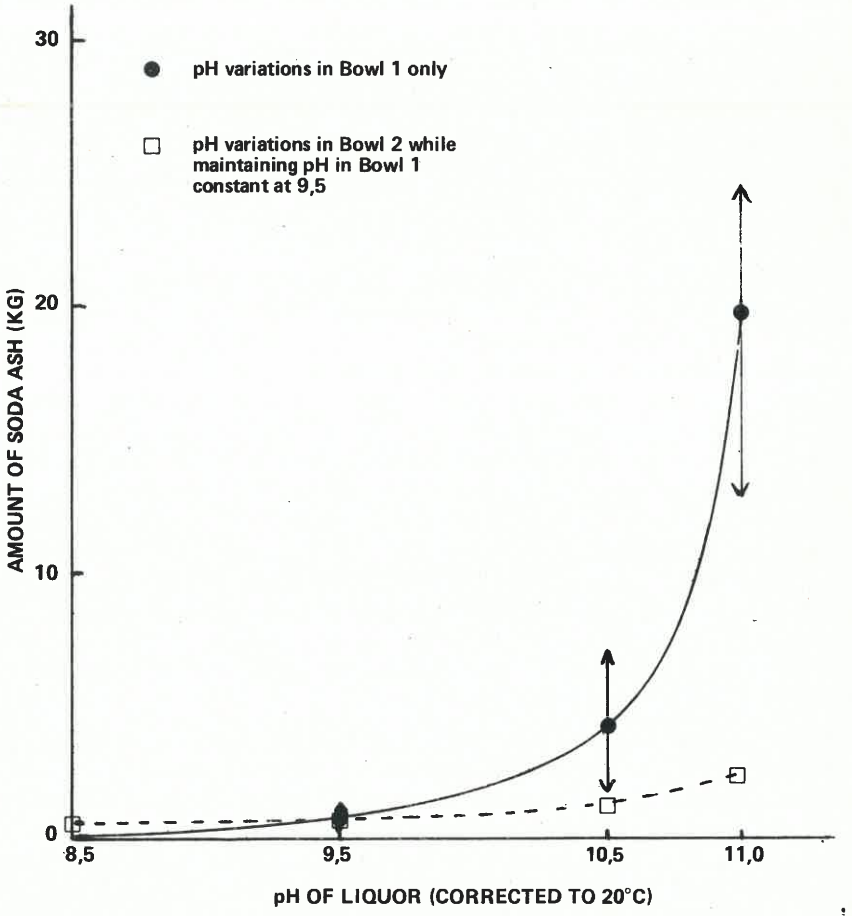

FIGURE 1

Total soda ash additions for various pH values of the liquors

TABLE I

AMOUNTS OF DETERGENT USED AND RESIDUAL GREASE LEVELS OBTAINED ON THE WOOL FOR VARIOUS pH AND TEMPERATURE VALUES OF THE LIQUOR IN THE FIRST SCOURING BOWL

pH OF LIQUOR IN BOWL 1		8,5				9,5				10,5				11,0			
Temperature of Liquor in Bowl 1 (°C)		52	55	60	65	52	55	60	65	52	55	60	65	52	55	60	65
TOTAL DETERGENT USED (ml) (Initial charge plus supplementary additions)	Bowl 1	6000	3100	300	200	5800	1400	200	300	8500	800	200	0	2000	0	0	0
	Bowl 2	1700	1100	500	500	400	450	400	400	400	400	400	300	400	250	200	150
	Bowl 3	800	400	150	150	300	200	150	150	150	150	150	100	150	100	120	100
	Total	8500	4600	950	850	6500	2050	750	850	9050	1350	750	400	2550	350	320	250
Average Total		3725				2538				2888				868			
RESIDUAL GREASE ON WOOL (%)	After 1st bowl	4,1	1,5	4,6	6,2	2,3	2,0	5,2	4,8	1,3	1,0	2,0	5,8	2,3	1,3	3,1	3,4
	Average	4,1				3,6				2,5				2,5			
	After completion of scouring	0,88	0,24	0,24	0,22	0,94	0,38	0,43	0,35	0,51	0,20	0,12	0,30	0,39	0,19	0,38	0,30
	Average	0,40				0,53				0,28				0,32			
Dichloromethane extractable matter on wool after 3 gillings (%)		0,89	0,35	0,34	0,31	0,79	0,55	0,60	0,44	0,69	0,31	0,30	0,45	0,44	0,26	0,44	0,47
	Average	0,47				0,60				0,44				0,40			
pH of aqueous extract after 3 gillings		7,2	7,2	7,0	6,9	6,9	6,9	6,9	6,9	7,0	7,0	8,2	8,1	8,9	9,3	9,2	9,4
	Average	7,1				6,9				7,6				9,2			

TABLE II

AMOUNTS OF DETERGENT USED AND RESIDUAL GREASE LEVELS OBTAINED ON THE WOOL FOR VARIOUS pH VALUES OF THE LIQUOR IN THE SECOND SCOURING BOWL

pH of Liquor in Bowl 1		9,5	9,5	9,5	9,5
pH of Liquor in Bowl 2		8,5	9,5	10,5	11,0
Detergent added (ml)	Bowl 1	1400	1400	1400	1400
	Bowl 2	450	300	550	350
	Bowl 3	200	150	200	150
	TOTAL	2050	1850	2150	1900
Residual grease on scoured wool (%)	After 1st Bowl	2,0	2,5	2,9	2,5
	After completion of scouring	0,38	0,33	0,51	0,32
pH of aqueous extract after 3 gillings		6,9	7,6	8,2	9,8

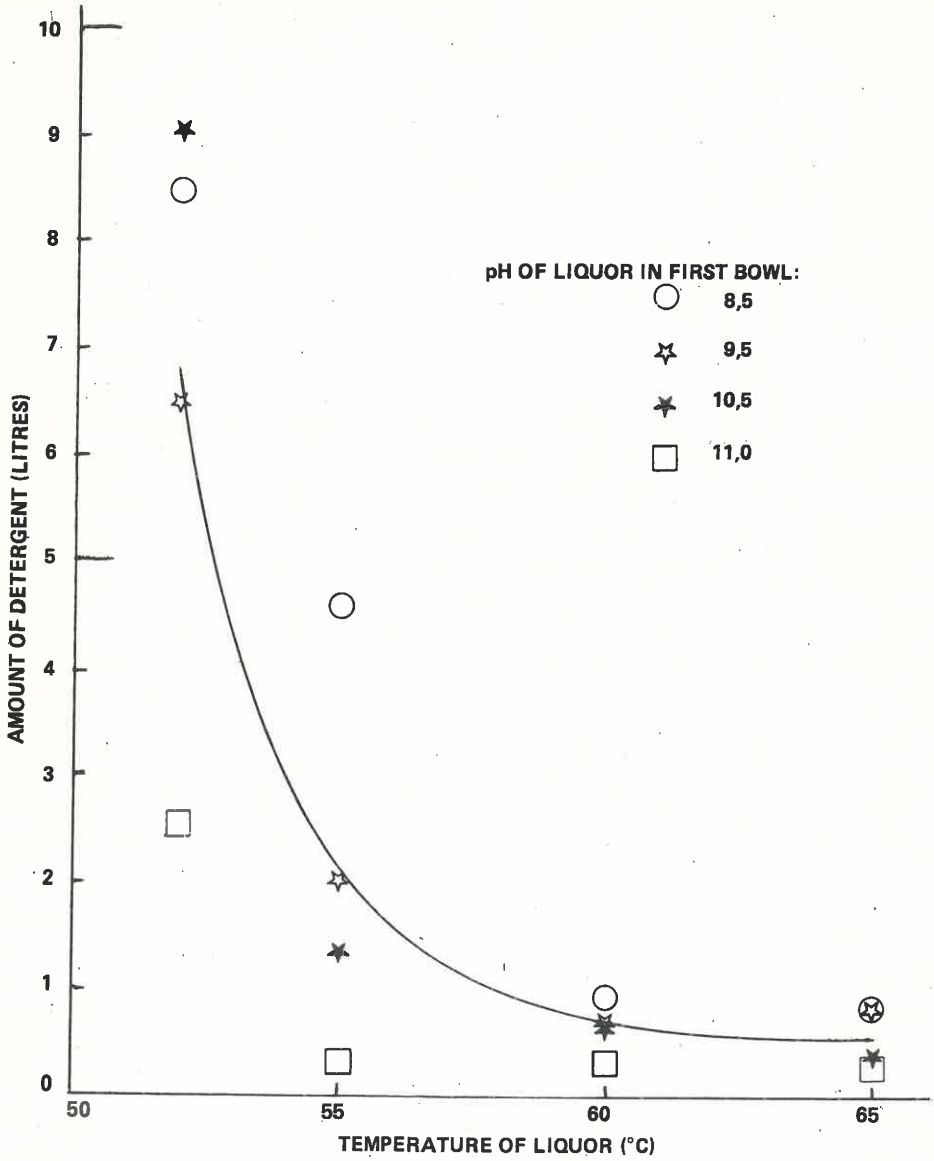

FIGURE 2

Total amount of detergent used versus temperature of the liquor in the first bowl

It can be seen from Table I that the residual grease on the wool emerging from the first bowl varied widely from about one to six *per cent. Average* values for the different pH values varied from 2,5 to 4,1 which appeared more satisfactory. The residual grease levels on the wool after completion of scouring were difficult to regulate in those cases where the temperature of the liquor in the first bowl was 52°C, particularly when the pH was relatively low. It is clear from Table I that to have matched the residual grease results obtained at the higher temperatures considerably more detergent should have been used when scouring at this temperature. Apart from the results for 52°C all others, with one exception, lay between 0,2 and 0,4 *per cent* after scouring and the dichloromethane extractable matter lay between 0,3 and 0,6 *per cent* after gilling. *Average* values for different specific pH values varied from 0,3 to 0,5 *per cent* after scouring (residual grease), and from 0,4 to 0,6 *per cent* after gilling (dichloromethane extractable matter) which appeared more satisfactory.

From Table II it is clear that when the pH of the liquor in the *second* bowl was varied from 8,5 to 11,0 while keeping the pH of the liquor in the first bowl constant at 9,5 the amounts of detergent used were, to all intents and purposes, constant in each case. Residual grease levels on the wool emerging from the first bowl were within the relatively narrow range of two to three *per cent* and, after scouring had been completed, were within the range of 0,3 to 0,5 *per cent*. These results were considered satisfactory.

Variations in the results for residual grease, which have been discussed above, illustrate the difficulties encountered during the experiment. While it has been mentioned that most of the results were satisfactory it would, nevertheless, seem wise to consider essentially average, overall trends, and to exercise particular caution in trying to interpret individual results.

There were no neps $>1,0$ mm in diameter observed in the card slivers during any of the experiments undertaken. The number of neps $>0,5$ but $\leqslant 1,0$ mm were very few indeed and varied in the range 0 to 8 per 20 g. These were so few that it was not possible to expect nor to find a correlation with any of the parameters measured. The results for this size of nep have therefore been added to those for neps $\leqslant 0,5$ mm (which were predominant) for the purpose of plotting the figures. In Fig. 3 the results have been plotted against the pH of the liquor and in Fig. 4 the results have been plotted against the temperature of the liquor.

The average trend for the number of neps after carding when the pH of the liquor in the *first* scouring bowl was varied from 8,5 to 11,0 is depicted by the solid line in Fig. 3. It is quite clear that the number of neps showed a trend towards increasing as the pH of the liquor increased. The spread of values obtained at the different specific pH values is also shown, and appears largest at pH 10,5. In Fig. 4 an attempt has been made to show tendencies at each specific pH to demonstrate more clearly than is evident from Fig. 3 that, when soda ash was used, the tendency was for neps to increase as the temperature of the first bowl was increased. Conversely, when *no* soda ash was used an increase in temperature appeared to

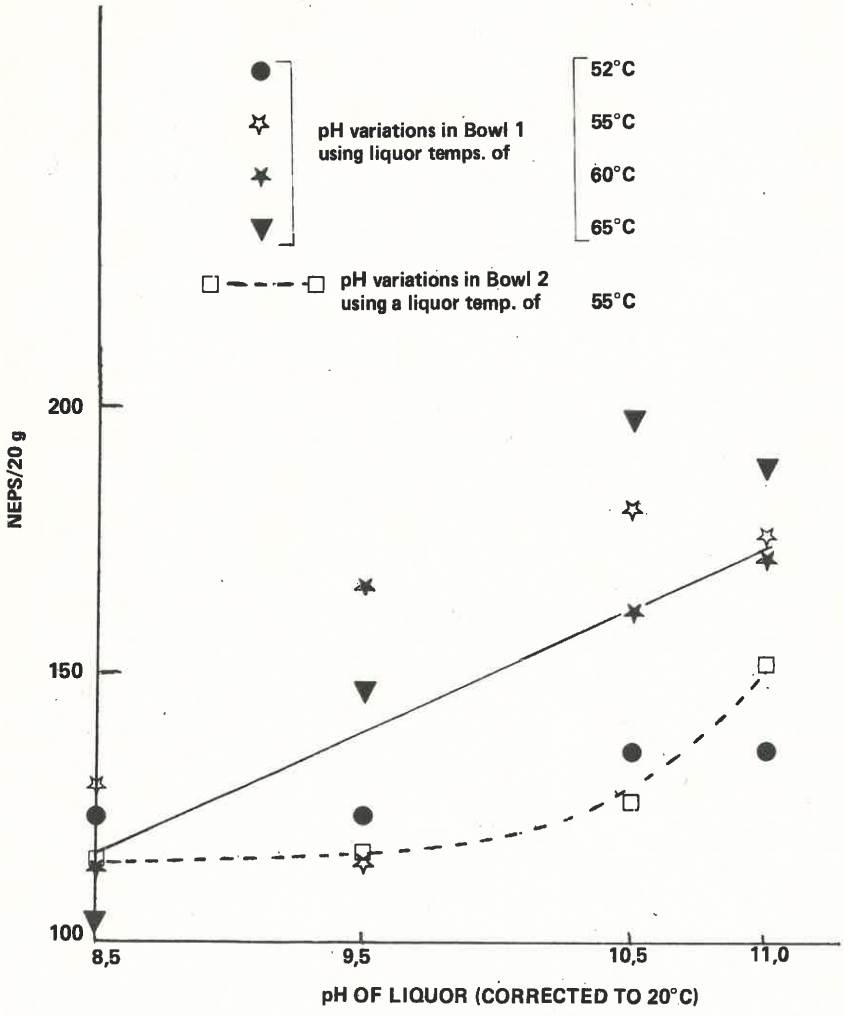

FIGURE 3

Total number of neps after carding versus pH of the liquor

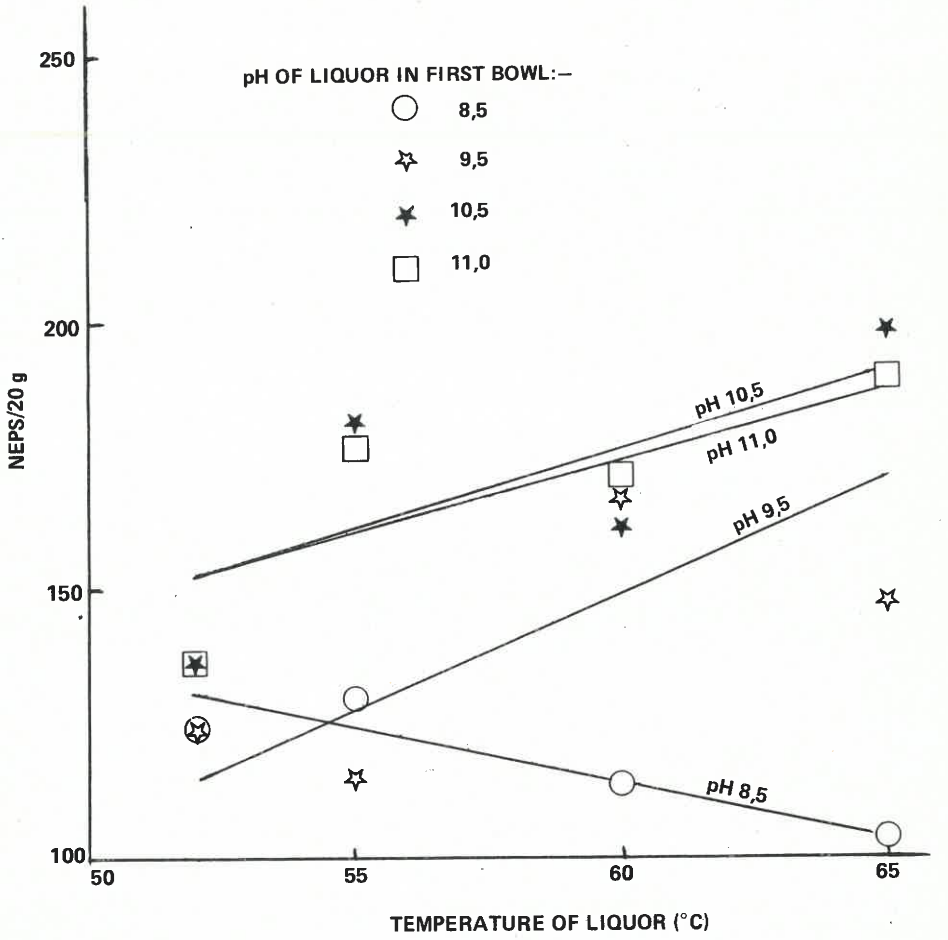

FIGURE 4

Total number of neps after carding versus temperature of the liquor in the first bowl

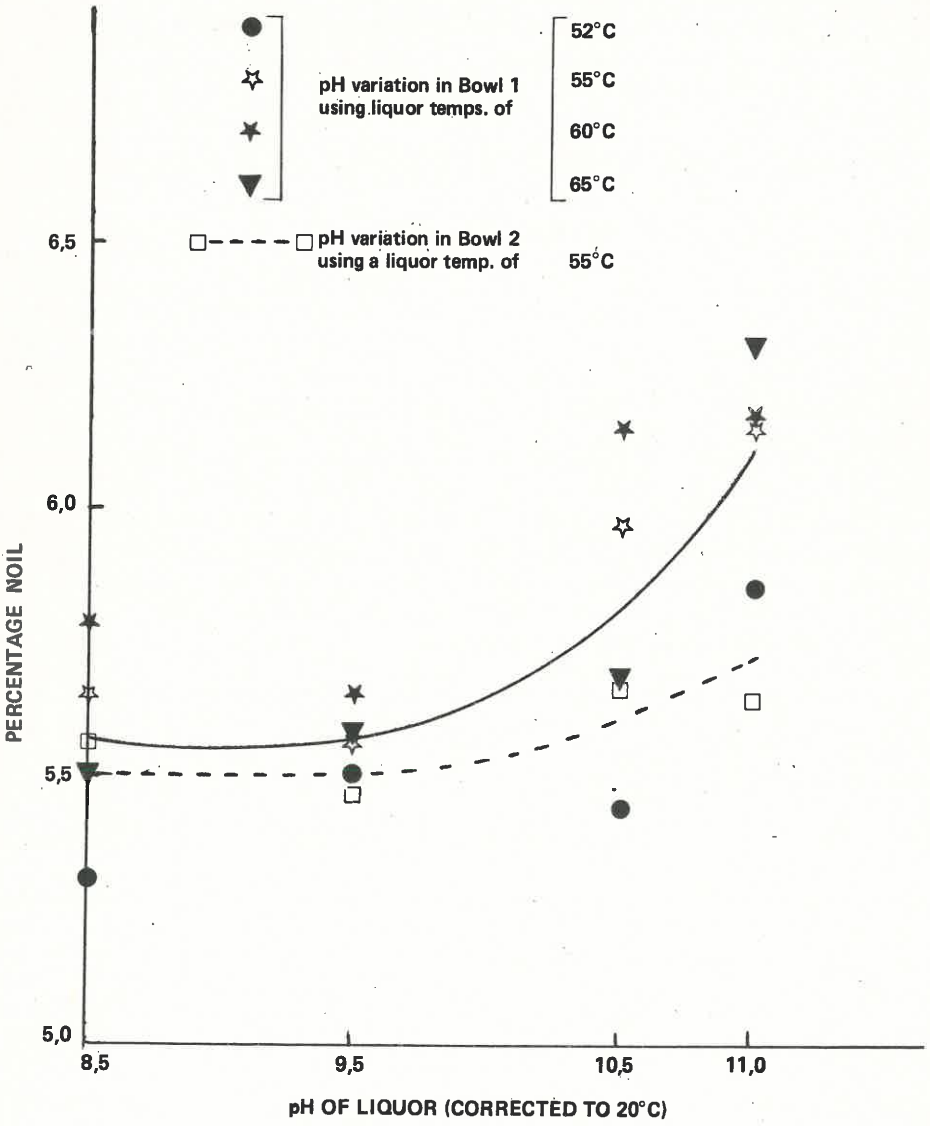

FIGURE 5

Percentage noil during combing versus pH of the liquor

result in a reduction in the nep count (in the range 52°–65°C).

The broken line in Fig. 3 indicates the trend for the number of neps after carding when the pH of the liquor in the second scouring bowl was varied from 8,5 to 11,0 and the temperature of the liquor was 55°C. It would seem that the nep count tended to be lower for a specific pH value of the liquor in the *second* bowl than it was for that same value when the *first* bowl was used at the same temperature. In addition, the broken line shows a more gradual slope up to a pH value of 10,5 indicating that the detrimental effect of increasing pH on the nep count was not so marked as in the previous case.

The average trend for the percentage noil obtained during combing when the pH of the liquor in the *first* scouring bowl was varied from 8,5 to 11,0 is depicted by the solid line in Fig. 5. The actual points illustrate the spread of values obtained at the different pH values. The spread of values was quite large particularly at pH 10,5. It would seem, however, that percentage noil tended to remain fairly constant from pH 8,5 to 9,5 and to increase as the pH was increased above this value.

The broken line in Fig. 5 indicates the trend for percentage noil when the pH of the liquor in the *second* scouring bowl was varied from 8,5 to 11,0 and the temperature of the liquor was 55°C. It would seem that percentage noil under these conditions tended to be lower than the values obtained when the pH of the liquor was adjusted in the *first* scouring bowl at the same temperature. The slope of the line also indicates that the detrimental effect of increasing pH on the percentage noil was not so marked as in the previous case.

Liquor pH values in the first bowl of up to 10,5 had hardly any effect on the pH of the aqueous extract of the wool (see Table I). When the pH was adjusted in the second bowl, however, the effect was more marked, the pH of the aqueous extract of the wool increasing gradually from 6,9 at a liquor pH of 8,5 to 9,2 at a liquor pH of 11,0 (see Table II).

SUMMARY AND CONCLUSIONS

Experiments were carried out in an attempt to determine the effect of liquor pH and temperature during conventional grease wool scouring to a residual grease level of approximately 0,4 *per cent* on the nep count of carded slivers produced. The percentage noil produced during subsequent combing was also studied.

Two series of experiments were carried out. In the first series four different pH values (from 8,5 to 11,0) of the liquor in the *first* bowl were used. In the second series the pH of the liquor in the first bowl was kept constant at 9,5 but the pH of the liquor in the *second* bowl was varied from 8,5 to 11,0. The experiments in the second series were carried out at constant temperature.

The amounts of wool used for each experiment were small, and no repeat experiments were undertaken. The amounts of soda ash and detergent used were not considered commercial because of the short duration of the experiments.

Although an attempt was made to obtain similar scouring efficiencies in all bowls in every experiment, some wide fluctuations were to be expected under the widely differing conditions and did in fact materialise. Judgement of the effects of changes in pH and temperature was, therefore, based in favour of average overall trends rather than individual results.

Excessive amounts of soda ash were required to maintain high pH values in the *first* scouring bowl, due possibly to the buffering action of the suint whereas significantly lower amounts were required to maintain the same values in the *second* bowl. Relatively small amounts of soda ash were required to maintain pH values of 9,5 in either the first or both the first and second scouring bowls. The amount of detergent required generally decreased as the pH of the liquor increased, but at 60° and above the effect of pH appeared negligible and the amounts of detergent used were small.

The number of neps after carding tended to increase with increasing liquor pH and also with increasing liquor temperature when soda ash was used. When no soda ash was used an increase in the temperature of the liquor appeared to result in a reduction in the number of neps. It appeared that less neps were obtained when the pH of the liquor in the *second* bowl was adjusted than when the pH of the liquor in the *first* bowl was adjusted to the same specific values. The reason for this may be that a significantly lower concentration of soda ash was needed in the former case for the same specific pH value.

Percentage noil tended to remain fairly constant when the pH of the liquor was 9,5 or lower but to increase as the pH was increased above this value. It appeared that lower values for percentage noil were obtained when the pH of the liquor in the *second* bowl was adjusted than when the pH of the liquor in the *first* bowl was adjusted to the same specific values. This was probably mainly as a result of the lower nep count values. The effect of the pH of the liquor on the pH of the aqueous extract of the gilled sliver was slight when the pH was adjusted in the first bowl, but increased more rapidly with an increase in liquor pH when the pH was adjusted in the second bowl.

For optimum values of neps and percentage noil it would seem that *if soda ash were used* the pH of the liquor in the *first* bowl must preferably not exceed 9,5 and the temperatures should be below 60°C. If the resulting pH values of the aqueous extract of the scoured wool are too low, however, it would seem preferable, from both an economical as well as a processing point of view, to adjust the pH in the *second* rather than the *first* scouring bowl. *If no soda ash were used* it would seem that the temperature of the liquor should preferably be 60/65°C.

In view of the small quantities of wool used in these experiments an elaboration on certain aspects of them is envisaged.

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THE USE OF PROPRIETARY NAMES

The fact that proprietary names have been mentioned in this report does not in any way imply that SAWTRI recommends them or that there are not substitutes which may be of equal value or even better.

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