

STUDIES ON SOME BASIC ASPECTS OF UTILIZATION OF SUINT RECOVERED FROM PAKISTANI WOOLS

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(Received August 17, 1968)

Suint contents of four of the leading Pakistani carpet wools and also of waste scouring liquors from the mills were determined. The suint obtained was calcined and analysed for the major basic ions (potassium, sodium and calcium) and the acidic ions (sulphate, chloride, silicate, phosphate and carbonate). Attempts were made to crystallize fractions from suint, as an alternative procedure to calcination, aiming at the utilization of the suint. Hydrochloric acid was found to be more effective than sulphuric acid in facilitating crystallization and the optimum pH lay between 2 to 3.

Introduction

In order to remove the various impurities, raw wool is subjected to scouring, as one of the initial steps, in both woollen and top-making processes. The waste scouring liquors thus contain grease and suint in addition to dirt particles. The grease is recovered in most leading industrial areas, whereas suint is rarely isolated for further utilization on industrial scale. This part being rich in potassium salts can be used as a source for potassium fertilizers.

A preliminary survey indicated that carpet type of wool grown in such tropical areas as West Pakistan was poor in grease content but, on the other hand, normal or rich in suint content. The recovery of suint, therefore, could well be an economical proposition in such areas.

It may be pointed out that suint, as most biological products, is not uniform in composition, its components may vary from fleece to fleece¹ and its characteristics, such as pH², may be influenced by the various environmental and physiological factors. On an overall basis, it is a complex substance comprising a number of organic and inorganic components. In fact, beginning with Vauquelin's work in 1802,³ volatile and non-volatile fatty acids, some dicarboxylic acid, nitrogenous materials which may include peptides, urea, amino acids, nitrogenous bases and possibly phenols, certain neutral materials, carbohydrates, and potassium as the major inorganic ion associated with the acidic constituents have been identified to be present in variable quantities as summarised by Howitt and Preston.⁴ On a quantitative basis, however, the composition may be put to be roughly as (i) inorganic anions and cations 56%, (ii) ampholytes 3%, (iii) neutral components 5%, and (iv) organic acids 34%.⁵

In a contemporary study,⁶ some of the basic aspects of the recovery of wool grease in Pakistan have been investigated. The present work was concerned with some of the important issues of recovery of potassium salts from the waste scouring liquors in the country. The objectives of this work were thus:

- (a) to scan the suint contents of some of the typical wool types in Pakistan as well as of the waste scouring liquors, obtaining as well as of the waste scouring liquors, obtaining from the mills.
- (b) to analyse the above samples of suint for their major components, and
- (c) to investigate some of the important aspects of economical recovery of potassium-rich fractions from the indigenous suint.

Materials and Methods

Wool Samples.—Samples of four leading indigenous wool types (Hashtnagri, Kaghani, Makrani and Michni) were employed. In addition scouring liquors from a large scouring train at Karachi as well as from a woollen mill up-country were also included in the study. The latter have been described frequently as 'commercial' samples in this paper.

Extraction and Incineration of Suint.—The wool sample was opened out manually and dusted to remove as much extraneous matter as possible. This was followed by extraction with distilled water at room temperature. In order to ensure complete removal of the suint, washing was repeated (3-5 times) till a clear extract was obtained. The extracts were combined and concentrated by evaporation at a metal plate heater. The suint

matter so obtained was transferred to a crucible and burned completely by means of a bunsen burner. The ashes were extracted with distilled water, filtered and the filtrate evaporated to dryness. This was followed by dissolving in distilled water again, adding a little charcoal, filtering, concentrating and crystallising.

The commercial samples were first cracked with sulphuric acid by a procedure similar to Edwards *et al.*,⁷ where the suint is obtained as the filtrate, which is subsequently treated as above.

Determination of the Major Components.—Preliminary investigations revealed that the major basic ions present were potassium, sodium and calcium. Attention was, therefore, focussed on these elements in accordance with the objectives of the study.

- (a) The amounts of potassium, sodium and calcium radicals were determined by a Beckman Model B Spectrophotometer employing the usual procedures.⁸ The determination of sodium and potassium was repeated gravimetrically,⁹ and that of calcium titrimetrically¹⁰ and also by using EDTA.¹¹
- (b) Chlorides¹² and sulphates¹³ were determined gravimetrically.
- (c) Phosphates were determined by the usual gravimetric methods¹⁴ and also by the colorimetric¹⁵ procedure employing molybdenum blue reaction.
- (d) Silicates were determined colorimetrically.¹⁶
- (e) Carbonates were determined titrimetrically.¹⁷

Recovery of Salts.—In the rather limited number of instances where suint has been recovered on a commercial basis, the method adopted seems to have been, in principle, that of calcination. This is an expensive unit process and would seem to limit the utilization of suint as a fertilizer, as a variety of rather inexpensive fertilizers is already available in the market. It was, therefore, considered desirable that some alternative economical procedures aiming at recovering the major components under suitable conditions be explored.

In a preliminary set of experiments suint was extracted from raw wool, employing distilled water in about 50:1 liquor to wool ratio. As a

result of concentration on heating a viscous mass resulted, in confirmation of Freney's results. On acidifying with sulphuric acid, to various pH levels, however, crystals usually deposited, although loaded heavily with organic matter. The solution was heated at the boil for varying intervals of time to effect specific changes in the gravity, and left to crystallize at room temperature. The mother liquor was heated to a specific concentration and allowed to recrystallize. Thus a number of fractions were obtained. These initial experiments indicated that pH was of significant importance in facilitating the crystallization and the optimum pH lay in the range 1–3. Experiments were, therefore, repeated under specific conditions with a view to identifying the optimum pH, more accurately. The conditions have been described elsewhere.

The preliminary experiments indicated that either mass crystallization of crude suint or effective fractional crystallization was probably not feasible within relatively a smaller number of steps. In order to be specific, therefore, a number of attempts were made at fractional crystallization, obtaining different number of fractions in each attempt. The difficulties in crystallization seemed to have been caused by the presence of the impurities as well as the tendency of the suint solution to deposit an insoluble material with the passage of time as also observed by Howitt and Preston.⁴ In order to facilitate crystallization, a few crystals of either potassium or sodium salts were added at the various stages; the conditions of crystallization have been described in the Results section. The mother liquor was shaken with ethyl alcohol, when the inorganic matter separated on centrifuging. This matter was subsequently considered as representing the suint matter of the mother liquor.

As crystallization with sulphuric acid seemed to be not effective enough to merit commercial feasibility, an alternative procedure, employing hydrochloric acid was considered worth investigation. A number of attempts at crystallization were made in this case as well, and the fractions obtained were subjected to the quantitative analyses.

Results and Discussions

Suint Content.—The percentage of solid suint in the raw wools as well as the waste scouring liquors is given in Table 1, which also includes the corresponding grease content and fineness.⁶ Sweeten¹⁸ and Lipson and Black¹⁹ reported similar data in respect of Merino and Corriedale wools which have been included for comparison.

in the lower half of the table. It is apparent that Pakistani wools, although very poor in grease content, are associated with usual suint content. Thus whereas in the Australian wools, the ratio of grease to suint content is about 2:1, it is about 1:3 in the case of Pakistani wools.

The incidence of low grease content in the indigenous wools has been discussed by Khan *et al.*⁶ By comparison, the high suint content (almost at par with that of foreign wools) indicates a lack of correlation between grease and suint contents. It may be pointed out that this high content does not appear to be a consequence of the hot climatic conditions as the suint is probably not sweat or perspiration produced to reduce temperature.²⁰ On the other hand, it appears that sheep widely differing in origin and kept

under different climatic conditions do not appreciably differ in their suint production.

It may be pointed out that the amounts of suint in the scouring liquors from the mills are not influenced at present by any consideration of the recovery of suint; and when such a consideration is given, the process could perhaps be regularized to facilitate optimum recovery.

Results of analysis of the suint ash are given in Table 2. With the exception of commercial samples, duplicate samples were tested in each case, and the results of basic ions obtained by photometric method were confirmed by those obtained with other techniques as mentioned previously. The values indicate large variation in the percentage of various constituents from

TABLE 1.—SUINT CONTENT OF WOOL (%).*

| Type of Wool | Oven dry Washed wool | Dirt | Moisture | Suint | Grease** | Fineness** (μ) | Remarks |
|--|----------------------|-------|----------|-------|----------|----------------|--|
| Hashtnagri | 60.70 | 18.50 | 10.6 | 6.25 | 2.6 | 46.2 | Crystallized fractions added up. Material not calcined |
| " | 62.69 | 15.80 | 10.6 | 7.55 | | | |
| " | 62.90 | 15.40 | 10.6 | 7.12 | | | |
| " | 61.50 | 16.20 | 10.6 | 7.90 | | | |
| " | 59.40 | 18.20 | 10.4 | 5.15 | 2.6 | 46.2 | Calcined |
| Michni | 63.70 | 11.46 | 12.0 | 6.48 | 2.2 | 49.5 | " |
| " | 63.98 | 12.01 | 12.0 | 7.81 | | | |
| Makrani | 61.70 | 15.75 | 11.5 | 4.57 | 4.00 | 40.4 | " |
| " | 62.25 | 15.30 | 11.5 | 5.12 | | | |
| Kaghani | 62.20 | 14.74 | 12.2 | 4.55 | 4.5 | 39.6 | " |
| " | 60.65 | 15.29 | 12.3 | 6.10 | | | |
| Commercial 1st tank from Mill No. 1 | — | — | — | 0.696 | } | | % on volume basis |
| Commercial 2nd tank from Mill No. 1 | — | — | — | 0.447 | | | |
| Commercial from Mill No. 2, counter current method | — | — | — | 0.614 | | | |
| Merino | 50.20 | 18.40 | 9.8 | 6.0 | 15.0 | | |
| Crossbred | 60.20 | 8.20 | 11.9 | 7.6 | 11.8 | | |

*Calculated on the basis of raw wool at 65% R.H. and 21°C except as indicated.

**These values are the average for the breed.⁶

TABLE 2.—ANALYSIS OF SUINT ASH (%).

| Type of wool | Carbonate | Chloride | Sulphate | Silicate | Phosphate | Potassium | Sodium | Calcium |
|---|-----------|----------|----------|----------|-----------|-----------|--------|---------|
| Michni | 30.95 | 3.54 | 2.87 | 1.20 | 2.60 | 50.25 | 3.99 | 2.45 |
| Makrani | 24.08 | 2.19 | 2.30 | 2.00 | 1.33 | 61.80 | 3.25 | 2.65 |
| Kaghani | 29.92 | 2.74 | 3.25 | 3.03 | 1.06 | 45.72 | 4.85 | 3.67 |
| Hashtnagri | 24.43 | 3.84 | 3.38 | 2.85 | 1.99 | 55.38 | 3.89 | 2.55 |
| Commercial 1st tank from Mill No. 1 | — | — | 30.43 | — | — | 38.38 | 4.20 | 8.25 |
| Commercial 2nd tank from Mill No. 1 | — | — | 30.04 | — | — | 33.42 | 4.60 | 8.36 |
| Commercial from Mill No. 2 and (counter-current method) | — | — | 28.62 | — | — | 37.38 | 4.00 | 9.35 |

TABLE 3.—FRACTIONS WITH SULPHURIC ACID.

| Sample No. | Wt of wool (g) | pH | | Fractions | Volume | | Specific gravity | | Time | | Yield in (g)* | | Yield %* | | Yield from un-crystallizable matter by the addition of ethyl alcohol | | | | |
|------------|----------------|---------|-------|-----------|---------|-------|------------------|--------|----------------|---------|---------------|--------|---------------|---------|--|------|--|--|--|
| | | Initial | Final | | Initial | Final | Initial | Final | Heating hr min | Cooling | Each fraction | Total | Each fraction | Overall | g | % | | | |
| I | 50.8 | 8.5 | 2-3 | 1st | 1000 | 595 | 1.0003 | 1.0149 | 1 45 | 24 | 0.2316 | | 0.46 | | | | | | |
| | | | | 2nd | 595 | 325 | 1.0149 | 1.0628 | 1 15 | 24 | 0.6838 | | 1.36 | | | | | | |
| | | | | 3rd | 325 | 10 | 1.0628 | — | 2 0 | 24 | 1.3289 | 2.1443 | 2.43 | 7.12 | 1.3234 | 2.62 | | | |
| II | 49.45 | 8.8 | 1-2 | 1st | 1000 | 442 | 1.0072 | 1.0542 | 2 0 | 24 | 0.2201 | | 0.43 | | | | | | |
| | | | | 2nd | 442 | 376 | 1.0542 | 1.0230 | 0 20 | 24 | 0.0253 | | 0.05 | | | | | | |
| | | | | 3rd | 376 | 122 | 1.0230 | 1.0388 | 1 20 | 24 | 0.6686 | | 1.32 | | | | | | |
| | | | | 4th | 122 | 65 | 1.0388 | 1.0544 | 0 18 | 24 | 0.5689 | | 1.12 | | | | | | |
| | | | | 5th | 65 | 10 | 1.0544 | — | 0 40 | 24 | 0.8839 | 2.3667 | 1.75 | 7.55 | 1.2907 | 2.56 | | | |
| III | 73.65 | 8.5 | 1-2 | 1st | 1410 | 1050 | 1.0072 | 1.0177 | 1 55 | 24 | 0.0997 | | 0.13 | | | | | | |
| | | | | 2nd | 1050 | 1020 | 1.0177 | 1.0102 | 0 16 | 24 | 0.5383 | | 0.72 | | | | | | |
| | | | | 3rd | 1020 | 790 | 1.0102 | 1.0103 | 1 15 | 24 | 0.7195 | | 0.97 | | | | | | |
| | | | | 4th | 790 | 620 | 1.0103 | 1.0178 | 0 50 | 24 | 0.6624 | | 0.89 | | | | | | |
| | | | | 5th | 620 | 398 | 1.0178 | 1.0410 | 1 0 | 24 | 0.5346 | | 0.72 | | | | | | |
| | | | | 6th | 398 | 155 | 1.0410 | 1.0455 | 1 0 | 24 | 0.5923 | | 0.78 | | | | | | |
| | | | | 7th | 155 | 10 | 1.0455 | — | 0 40 | 24 | 0.7560 | 3.9028 | 1.02 | 7.90 | 1.9223 | 2.59 | | | |

*Calculated on the basis of raw wool.

TABLE 4.—ANALYSIS OF VARIOUS FRACTIONS FROM TABLE 3 (%).

| Sample | Fraction | K | Na | Ca | K ₂ SO ₄ | Na ₂ SO ₄ | CaSO ₄ | Overall percentage in each sample | | | |
|--------|---------------|-------|-------|-------|--------------------------------|---------------------------------|-------------------|-----------------------------------|--------|---------------------------------|--------|
| I | 1st | 0.08 | 0.008 | 21.24 | 0.18 | 0.03 | 72.22 | K ₂ SO ₄ | =60.10 | | |
| | 2nd | 8.04 | 0.02 | 11.79 | 17.82 | 0.67 | 35.45 | | | Na ₂ SO ₄ | = 8.47 |
| | 3rd | 30.43 | 2.98 | 0.14 | 67.83 | 6.13 | 0.25 | | | CaSO ₄ | = 7.02 |
| | Mother liquor | 28.25 | 2.20 | — | 53.16 | 6.80 | — | | | | |
| II | 1st | 0.12 | 0.02 | 21.48 | 0.26 | 0.06 | 73.04 | K ₂ SO ₄ | =60.80 | | |
| | 2nd | 0.12 | 0.04 | 20.08 | 0.26 | 0.13 | 68.25 | | | Na ₂ SO ₄ | = 8.63 |
| | 3rd | 10.72 | 0.23 | 5.92 | 24.64 | 0.72 | 20.13 | | | CaSO ₄ | = 6.13 |
| | 4th | 20.54 | 1.09 | 0.94 | 45.88 | 3.37 | 3.33 | | | | |
| | 5th | 28.44 | 1.24 | 0.07 | 63.19 | 3.83 | 0.25 | | | | |
| | Mother liquor | 24.33 | 1.64 | — | 54.23 | 5.08 | — | | | | |
| III | 1st | 0.18 | 0.00 | 20.50 | 0.05 | 0.00 | 74.42 | K ₂ SO ₄ | =54.40 | | |
| | 2nd | 0.18 | 0.84 | 14.82 | 0.40 | 0.23 | 40.35 | | | Na ₂ SO ₄ | = 6.30 |
| | 3rd | 10.25 | 0.17 | 6.15 | 22.80 | 1.79 | 20.89 | | | CaSO ₄ | = 6.20 |
| | 4th | 12.52 | 0.30 | 2.82 | 27.60 | 1.89 | 9.60 | | | | |
| | 5th | 24.52 | 0.39 | 0.36 | 54.27 | 1.93 | 1.20 | | | | |
| | 6th | 27.24 | 1.76 | 0.08 | 60.52 | 7.44 | 0.29 | | | | |
| | 7th | 29.22 | 1.43 | 0.08 | 64.90 | 4.42 | 0.26 | | | | |
| | Mother liquor | 24.92 | 1.45 | — | 55.33 | 4.48 | — | | | | |

TABLE 5.—ANALYSIS OF RECRYSTALLIZED POTASSIUM-RICH FRACTIONS FROM TABLE 4.

| Sample | Fraction | K | Na | Ca | K ₂ SO ₄ | Na ₂ SO ₄ | CaSO ₄ |
|--------|---------------|-------|------|-------|--------------------------------|---------------------------------|-------------------|
| I | 3rd | 38.36 | 2.04 | 1.05 | 85.24 | 6.24 | 3.58 |
| | Mother liquor | 29.65 | 2.30 | — | 66.08 | 8.01 | — |
| II | 4th | 29.04 | 1.99 | 1.50 | 64.42 | 6.14 | 5.10 |
| | 5th | 36.41 | 1.72 | 0.44 | 80.88 | 5.33 | 1.52 |
| III | Mother liquor | 30.01 | 2.82 | — | 63.55 | 8.74 | — |
| | 7th | 39.92 | 1.88 | 0.08 | 88.8 | 5.73 | 0.26 |
| | Mother liquor | 29.99 | 2.36 | 0.088 | 65.85 | 7.25 | 0.02 |

sample to sample. It may, however, be concluded that the suint ash from water extracts of Pakistani wools contains 40–60% potassium, 4–5% sodium and 2–3% calcium. This is in general conformity with the available reports,^{3,4} although the appreciable proportions of calcium and sodium are more in agreement with the recent result.⁴ The suint recovered from the scouring liquors from the mills has slightly different composition, the amounts of sodium and calcium being still higher.

Crystallization with Sulphuric Acid.—The amounts of the various fractions obtained are given in Table 3 and the results of their analysis in Table 4.

Suint solutions, acidified with sulphuric acid differed in their behaviour from the unacidified

ones only slightly. Calcium sulphate being insoluble, however, separates readily. Evaporating the solution to high concentration did not result in effective crystallization, and, if at all, a few crystals heavily superimposed by viscous matter, were obtained. Evaporating to lower concentration, followed by removal of the mother liquor, however, led to fractional crystallization. Nevertheless, it is evident from Table 3 that crystallization is very sluggish under these conditions and from Table 4 that potassium does not effectively crystallize as sulphate even in the last fraction, a substantial amount passing through to the mother liquor, left over at the end. Although a change of conditions to pH 2–3 instead of 1–2 proved to be helpful, yet, in either case and even after obtaining seven fractions, about one third of the total potassium passes through to the mother liquor.

The rather sluggish crystallization of potassium sulphate is understandable on the basis of its solubility data,²¹ according to which it is about three times as much soluble as sodium sulphate at the identical temperatures. The temperature of crystallization selected in this study was about 10°C. However, almost negligible crystallization of this salt from the mother liquor, left over at the end, is probably due to interfering effects of other substances present, especially of the organic type which get more and more concentrated in the liquor as the inorganic matter is crystallized out at the various stages.

Crystals from some of the fractions were re-dissolved in distilled water, recrystallized, and subjected to analysis. The results are given in Table 5, which shows marked improvement in the purity of the crystals.

In view of the above difficulties, it was sought to facilitate crystallization by introducing a few crystals of the suitable types, as detailed in Table 6. The amounts of the fractions obtained are given in the same table and the results of their analyses in Table 7. By comparison with Tables 4 and 5, the addition of crystals has been of some help but the process seems to be still too cumbersome to merit practical utility.

These conditions naturally led to the conclusion that sulphuric acid, as an agent in the suint recovery, can hardly prove promising. An alternative approach, employing hydrochloric acid, was, therefore, adopted as described earlier.

Crystallization with Hydrochloric Acid.—The amounts of the various fractions obtained are given in Table 8, and the results of their analyses in Table 9.

It can be seen from these tables that, in comparison to sulphuric acid, hydrochloric acid is a far more suitable agent and readily effects mass crystallization of the inorganic material. With the introduction of the extra crystals to the liquor, the situation is altered very little in terms of the composition of the fractions obtained, but crystallization is greatly facilitated.

It can, therefore, be seen that employing small quantities of hydrochloric acid, mass crystallization can be effected (either with or without the added crystals) in a process involving relatively fewer number of steps, *viz.* 1–3. The mass so crystallized can be roughly described as comprising mainly 50–80% KCl, 7–10% NaCl and 2–5% CaCl₂.

 TABLE 6.—FRACTIONS BY THE ADDITION OF EXTRA CRYSTALS (WITH H₂SO₄).

| Sample | Wt of wool (g) | pH | | Frac-tion | | Volume | Sp. gr | Time | | Yield (g) | | Yield % | | Extra crystals | |
|--------|----------------|---------|-------|-----------|-------|--------|--------|----------------|---------|---------------|--------|---------------|---------|---|---|
| | | Initial | Final | Initial | Final | | | Heating hr min | Cooling | Each fraction | Total | Each fraction | Overall | | |
| I | 40.06 | 8.5 | 3-2 | 1st | 1000 | 580 | 1.0035 | 1 45 | 24 | 0.2002 | 0.43 | — | — | — | |
| | | | | 2nd | 580 | 62 | — | 1 15 | 24 | 0.5836 | — | 1.28 | — | — | — |
| | | | | 3rd | 62 | — | — | 1 45 | 24 | 1.8900 | 2.6740 | 4.08 | 5.99 | — | — |
| II | 50.10 | 8.5 | 3-2 | 1st | 1000 | 590 | 1.0557 | 1 45 | 24 | 0.2726 | 0.41 | — | — | — | |
| | | | | 2nd | 590 | 155 | — | 1 30 | 24 | 0.3020 | — | 0.60 | — | +0.308 g Na ₂ SO ₄ * | |
| | | | | 3rd | 155 | 45 | — | 2 0 | 24 | 1.3875 | — | 2.77 | — | +0.0103 g K ₂ SO ₄ * | |
| | | | | 4th | 45 | — | — | 2 0 | 24 | 1.6528 | 3.6129 | 3.33 | 6.60 | — | — |
| III | 50.10 | 8.8 | 3-2 | 1st | 1000 | 595 | 1.0557 | 1 45 | 24 | 0.2384 | 0.48 | — | — | — | |
| | | | | 2nd | 595 | 312 | — | 1 30 | 24 | 0.3316 | — | 0.68 | — | — | — |
| | | | | 3rd | 312 | 40 | — | 1 45 | 24 | 1.2213 | — | 2.46 | — | +0.0203 g K ₂ SO ₄ * | |
| | | | | 4th | 40 | — | — | 2 0 | 24 | 1.4238 | 2.9151 | 2.86 | 6.48 | +0.0402 g Na ₂ SO ₄ * | |

*These values are subtracted from those of corresponding fractions for calculating the percentage yield.

TABLE 7.—ANALYSIS OF FRACTIONS FROM TABLE 6 (%).

| Sample | Fraction | K | Na | Ca | K ₂ SO ₄ | Na ₂ SO ₄ | CaSO ₄ | Overall% in each sample |
|--------|----------|-------|------|-------|--------------------------------|---------------------------------|-------------------|---|
| I | 1st | 0.08 | 0.15 | 20.74 | 1.66 | 0.45 | 70.58 | K ₂ SO ₄ = 58.20 |
| | 2nd | 8.34 | 1.87 | 11.30 | 18.67 | 5.72 | 38.44 | Na ₂ SO ₄ = 12.50 |
| | 3rd | 21.08 | 2.87 | 0.07 | 69.08 | 6.85 | 0.26 | CaSO ₄ = 8.69 |
| II | 1st | 0.07 | 0.08 | 20.74 | 0.17 | 0.15 | 70.58 | K ₂ SO ₄ = 56.20 |
| | 2nd | 8.28 | 0.17 | 10.20 | 17.80 | 0.58 | 35.16 | Na ₂ SO ₄ = 12.20 |
| | 3rd | 16.55 | 1.84 | 0.98 | 36.85 | 5.62 | 2.15 | CaSO ₄ = 8.21 |
| | 4th | 33.28 | 3.98 | 0.03 | 73.99 | 12.35 | 0.10 | |
| III | 1st | 0.08 | 0.08 | 20.74 | 0.17 | 0.15 | 70.68 | K ₂ SO ₄ = 72.2 |
| | 2nd | 8.90 | 0.20 | 10.20 | 19.80 | 0.67 | 35.02 | Na ₂ SO ₄ = 12.2 |
| | 3rd | 24.55 | 1.55 | 0.79 | 54.75 | 4.89 | 2.07 | CaSO ₄ = 9.58 |
| | 4th | 36.55 | 2.58 | 0.20 | 81.16 | 7.92 | 0.78 | |

TABLE 8.—FRACTIONS BY THE ADDITION OF EXTRA CRYSTALS (WITH HCl).

| Sample | Wt of wool (g) | pH | | Frac-tions | Volume | | Sp. gr. | Time | | Yield (g) | | Yield % | | Extra crystals |
|--------|----------------|---------|-------|------------|---------|-------|---------|----------------|----------|---------------|--------|---------------|---------|-----------------|
| | | Initial | Final | | Initial | Final | | Heating hr min | Cool-ing | Each fraction | Total | Each fraction | Overall | |
| I | 65.6 | 8.5 | 2-3.5 | 1st | 2000 | 50 | 1.0035 | 6 | 0 | 24 | 1.6317 | | 2.50 | |
| | | | | 2nd | 50 | 10 | — | 0 | 10 | 24 | 1.8623 | | 2.53 | |
| | | | | 3rd | 10 | — | — | — | 24 | 1.6460 | 5.1400 | 2.51 | 7.83 | |
| II | 70.03 | 8.8 | 2-3 | 1st | 2000 | 25 | 1.0011 | 6 | 0 | 24 | 4.5432 | | 6.34 | +0.0432 g KCl* |
| | | | | 2nd | 25 | — | — | 0 | 10 | 24 | 1.2330 | 5.7762 | 1.99 | 8.22 |
| III | 73.97 | 8.5 | 2-3 | 1st | 2000 | 55 | 1.0035 | 6 | 0 | 24 | 5.6230 | | 7.60 | +0.1235 g KCl* |
| | | | | 2nd | 55 | 5 | — | 0 | 20 | 24 | 1.4545 | 7.0775 | 1.89 | 9.5 |
| IV | 75.6 | 8.8 | 2-3 | 1st | 2000 | 52 | 1.0035 | 6 | 0 | 24 | 4.7490 | | 5.87 | +0.3250 g NaCl* |
| | | | | 2nd | 52 | 5 | — | 0 | 20 | 24 | 2.4448 | 7.1738 | 3.70 | 9.7 |

* These values are subtracted from those of corresponding fractions for calculating the percentage yield.

TABLE 9.—ANALYSIS OF SAMPLES FROM TABLE 8(%).

| Sample | Samples | Na | K | Ca | NaCl | KCl | CaCl ₂ |
|--------|--|------|-------|------|-------|-------|-------------------|
| I | Selected crystals without introducing extra crystals. | 2.81 | 47.45 | 0.79 | 8.53 | 88.50 | 2.62 |
| II | Sample + extra KCl crystals crude as a whole. | 2.85 | 26.32 | 2.14 | 7.23 | 50.00 | 7.52 |
| III | Sample + extra KCl crystals. Recrystallized in distilled water. | 3.42 | 33.42 | 1.76 | 8.67 | 63.78 | 5.13 |
| IV | 2nd yield of crystals from 3rd above, after 24 hr. | 2.95 | 4.44 | 1.10 | 7.54 | 77.01 | 3.63 |
| V | Sample + extra NaCl crystals. Recrystallized in distilled water. | 3.70 | 36.66 | 1.43 | 8.13 | 69.03 | 4.75 |
| VI | 2nd yield of crystals from V, above after 24 hr. | 6.05 | 27.55 | 1.26 | 15.36 | 52.51 | 4.19 |

Conclusions

Pakistani carpet wools, although poor in grease content, are associated with usual suint content. The suint ash comprises mainly of potassium 50–60%, sodium 4–5% and calcium 2–3%.

Crystallization could be employed, as an alternative procedure to calcination, to obtain potassium-rich suint fractions as a source for potassium fertilizers.

Acidification with hydrochloric acid effectively facilitates crystallization, better than that with sulphuric acid. The optimum pH is 2–3. The crystals so obtained are roughly analogous to the suint ash in terms of their basic constituents.

Acknowledgement.—The authors are thankful to Mr. A.A. Wakil of these Laboratories for supplying some of the samples and also to the mills for supplying the scouring liquors. Thanks are also due to Mr. Mehdi Imam of the Forest Research Institute, Peshawar, for assistance in spectrophotometric analysis.

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