

TREATMENT AND UTILISATION OF COTTON WASTE FROM SPINNING MILLS

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SUMMARY

The possibilities of treating and utilising cotton waste or "cotton dust" in three ways are considered in this paper. A granular carbon possessing ion exchange properties was prepared from the waste by suitable treatment with sulphuric acid, sodium chloride and alum. The efficiency of the sulphonated carbon in removing calcium from 0.1% calcium chloride solution and fluorine from water treated with sodium fluoride was examined.

The waste material when powdered and incorporated into black alkaline soils (on which cotton is generally grown) treated with ammonium sulphate was found to control the loss of ammonia to a considerable extent from the soil system. This action of cotton waste was found similar to that of straw powder used under comparable conditions.

The powdered waste material when allowed to decompose along with other organic wastes was found to retain moisture in the system for a considerably long period, thus facilitating the decomposition of the materials to a greater extent.

Steeping the cotton waste in raw sewage remarkably hastened disintegration of the cotton material. By treatment of the cotton waste with sewage, adjusting the moisture content, the period of decomposition and other conditions, a valuable manure could be prepared for use in agriculture.

1. INTRODUCTION

Considerable quantities of cotton waste have to be disposed of daily from the cotton-spinning mills, *e.g.*, about 3 tons of waste material from a spinning mill in South India in which about 30 tons of cotton is processed every day. The waste or "dust" from certain spinning mills in South India has been found to consist mostly of waste cotton admixed with a relatively small amount of materials like the coverings of cotton seeds and particles of plant material.

A study of the literature on the subject would show that attempts have been made to treat cotton waste for making nitrocellulose (Landell, 1917),

waterproof composition (Lundberg, 1928), cotton linters (Dhingra and Mithel, 1949), rayon (Uno, 1934), hydrocellulose (Chelintsev and Izrailevskii, 1937), cellulose lacquer (Menesdorfer, 1949) and for recovering fats and waxes (Gronau, 1935). Other attempts to utilise the waste material include production of paper (Goncharov, 1935; Robinov, 1935; Cerini, 1948). At the same time it would appear that the cotton waste has so little market value, at any rate in India, that it is burnt and got rid of if it cannot be used as fuel or as manure. The possibility of treating and utilising the waste material on an economic basis and with special reference to Indian conditions was recently considered, and some of the experiments conducted in this connection are described in this paper. The experiments mainly relate to utilisation of the waste (a) for preparing an ion exchange material (b) for stabilising nitrogen in alkaline cotton soils fertilised with ammonium sulphate, and (c) for making organic manure along with other wastes.

2. MATERIAL AND METHODS

The material employed for the studies was obtained from a certain mill in South India through the kind courtesy of the late Dr. Gilbert J. Fowler. A representative and uniform sample of the bulk material for analytical purposes and for other laboratory studies was prepared by powdering a portion of it and passing it through 40-mesh sieve. The sample thus prepared had a dull brownish appearance; it was very fluffy (2.5 g. of the material when fairly closely packed in a glass tube occupied a volume of 10 c.c.); and on keeping, it was found to form small aggregates.

The results of chemical analysis of the cotton waste are given in Table I. The methods of analysis were mostly the same as those given in the *Chemists'*

TABLE I
Results of chemical analysis of the cotton waste

Composition	Percentages on air-dry basis*
Nitrogen (N)	0.97
Ether extract (fat)	3.78
Loss on ignition (organic matter)	85.26
Residue on ignition (mineral matter)†	14.74
Acid insoluble matter (mostly silica)	7.48
Iron and aluminium oxides (Fe_2O_3 & Al_2O_3)	2.76
Calcium (Ca)	1.12
Magnesium (Mg)	0.10
Sulphate (SO_4)	1.40

* The moisture content of the material was 7.61 per cent.

† A considerable portion of the mineral matter to be accounted for is potash.

Year Book (1936). Fluorine was determined by the method recommended by Sanchis (1934), and aluminium was determined by the method suggested by Hatfield (1934).

3. PREPARATION AND PROPERTIES OF THE ION EXCHANGE CARBON OBTAINED FROM COTTON WASTE

Preparation of the carbon.—The procedure adopted for the preparation of the carbon from the cotton waste was essentially the same as that adopted for the treatment of spent coffee grounds (Mohanrao and Pillai, 1954). 40 g. of the carbon (between 20- and 40- mesh sieves) was obtained when 100 g. of the cotton waste was treated with 150 c.c. of concentrated sulphuric acid (sp. gr. 1.84). It was interesting to note that a granular carbon could be obtained from a light fluffy cotton waste on treatment with sulphuric acid.

Efficiency of the carbon as a cation-exchanger.—20 g. of the carbon was taken in a glass percolating column of $1\frac{1}{8}$ inches diameter and its efficiency in removing calcium from calcium chloride solution was examined in the same way as was done in the case of the carbon from spent coffee grounds (Mohanrao and Pillai, *loc. cit.*). The observations are given in Table II.

TABLE II

Efficiency of the carbon prepared from cotton waste in removing calcium from 0.1% calcium chloride solution

Lot No.	Amount of calcium (mg. in 250 c.c.)									
	1st cycle		2nd cycle		3rd cycle		4th cycle		5th cycle	
	In the effluent	Taken up by the carbon	In the effluent	Taken up by the carbon	In the effluent	Taken up by the carbon	In the effluent	Taken up by the carbon	In the effluent	Taken up by the carbon
1	61.0	22.0	1.0	81.5	1.0	79.5	1.0	79.5	0.8	78.5
2	75.5	7.5	8.5	74.0	1.2	79.3	6.0	74.5	1.0	78.3
3	79.5	3.5	32.0	50.5	34.0	40.5	17.5	63.0	8.1	71.2
4	79.5	3.5	72.5	10.0	66.5	14.0	52.5	28.0	42.1	37.2
5	80.0	3.0	78.0	4.5	75.0	5.5	69.5	11.0	64.5	14.8
6	—	—	79.5	3.0	77.5	3.0	69.5	11.0	64.5	14.8
7	—	—	81.5	1.0	78.0	2.5	76.5	4.0	76.1	3.2
8	—	—	—	—	80.0	0.5	79.0	1.5	77.8	1.5
9	—	—	—	—	80.5	0.0	78.0	2.5	78.5	0.8
10	—	—	—	—	80.5	0.0	79.5	1.0	79.1	0.2
Blank	83.0	..	82.5	..	80.5	..	80.5	..	79.3	..
Total calcium taken up by the carbon	..	39.5	..	224.5	..	230.8	..	276.0	..	300.5

It may be observed from Table II that the efficiency of the carbon increased after repeated use from the 1st cycle to the 5th cycle. In the 5th cycle 20 g. of the carbon removed about 300 mg. of calcium, *i.e.*, 1 lb. of the carbon could remove about 17 g. of calcium as CaCO_3 . The bed was regenerated at the end of each cycle by treatment with 5% sodium chloride solution as indicated in the above procedure.

Efficiency of the carbon in removing fluorine from water.—20 g. of the carbon was taken in a glass percolating column of 7/8 inch diameter and its efficiency in removing fluorine from tap water, to which known amounts of sodium fluoride were added, was studied as in the case of the carbon from spent coffee grounds (Mohanrao and Pillai, *loc. cit.*). The results of the study are given in Table III.

TABLE III

Efficiency of the carbon prepared from cotton waste in removing fluorine from tap water to which sodium fluoride was added

Lot No.	Amounts of fluorine in the effluents (in p.p.m.)				
	1st cycle	2nd cycle	3rd cycle	4th cycle	5th cycle
1	0.7	0.6	0.4	0.4	0.3
2	>1.0	>1.0	0.8	0.8	0.4
3	>1.0	>1.0	>1.0	0.8	0.6
4	..	>1.0	>1.0	>1.0	0.7
5	..	>1.0	>1.0	>1.0	>1.0
Blank	2.4	2.4	2.4	2.4	2.4
Volume of water treated to non-toxic levels of fluorine (in litres)	1	1	2	3	4

The amounts of aluminium in the effluents were negligible.

The observations given in Table III would suggest that the efficiency of the carbon increased by repeated use. Thus in the 5th cycle 20 g. of the carbon treated 4 litres of water (*i.e.*, 1 lb. of the carbon could treat 20 gallons of water) before toxic amounts of fluorine came through.

4. LABORATORY STUDIES ON THE INFLUENCE OF COTTON WASTE ON THE LOSS OF AMMONIA FROM ALKALINE SOILS TREATED WITH AMMONIUM SULPHATE

It is known that from soils, especially alkaline soils, and on application of inorganic nitrogenous fertilisers such as ammonium sulphate, a considerable amount of nitrogen is lost in the form of ammonia which volatilises off. Attempts have been made to control this loss of nitrogen. In view of the earlier observations reported from this laboratory (Bhaskaran *et al.*, 1939) on the beneficial effect of cellulosic matter, *e.g.*, straw powder, on the conservation of biologically fixed nitrogen in soil, it was of interest to study the comparative influence of the cotton waste powder on the retention of nitrogen in the soil treated with ammonium sulphate.

Experiments were carried out with two typical black cotton soils, one from North India (the Punjab) and the other from South India (Guntur). The method employed for the estimation of ammonia volatilised from the soils is largely based on the method developed by Subrahmanyam (1937). The procedure consists in pasting a piece of filter-paper (previously acid treated, for example, Whatman No. 30 or 41) moistened with standard acid on the inside of a glass which just covers the vessel containing the soil; the Petri dish has been found suitable for this purpose. The ammonia evolved from the medium is absorbed by the acid. At convenient intervals the dish can be removed and the unused acid in the paper estimated by back titration against standard alkali after boiling to drive off carbon dioxide.

The observations on the extent of loss of ammonia from the two soils treated with the cotton waste over a period of 20 days are recorded in Tables IV and V. 25 g. lots of the soils (in Petri dishes) were employed for the experiments; the moisture content of the soil samples was maintained at 40% level (at room temperature).

The results given in Tables IV and V indicate that (*a*) a relatively small amount of nitrogen was lost in the initial stages from the soils treated with the cotton waste at two levels (20 and 40 tons per acre—0.5 g. of the waste per lb. of soil corresponds to 1 ton per acre), this loss however decreasing gradually, and (*b*) the presence of sufficient quantities of waste at these levels considerably reduced the loss of ammonia from ammonium sulphate added to the soils, the loss being reckoned on the difference between those occurring in the soil + ammonium sulphate + cotton waste and the soil + cotton waste only.

It may be observed from Tables IV and V that about 20 per cent. of nitrogen from the waste was lost in 20 days when the material was applied

TABLE IV

Influence of cotton waste on the stabilisation of nitrogen in the soil (the black cotton soil from Punjab) treated with ammonium sulphate

Treatment	2 days		6 days		12 days		16 days		20 days	
	N in mg.	N as %	N in mg.	N as %	N in mg.	N as %	N in mg.	N as %	N in mg.	N as %
1 Soil only	0.9	..	1.9	.	2.9	..	3.6	..	4.2	..
2 Soil+0.5 g. waste (20 tons/acre)	2.0	4.4	5.5	14.4	7.0	16.4	8.5	18.4	9.2	20.0
3 Soil+1.0 g. waste (40 tons/acre)	2.0	2.2	5.4	7.0	7.1	8.4	8.7	10.2	9.4	10.4
4 Soil+12.5 mg. AmSO ₄ (10 cwts./acre)	4.2	26.4	8.7	54.4	11.6	69.6	12.9	74.4	14.0	78.4
5 Soil+12.5 mg. AmSO ₄ +0.5 g. waste	5.3	26.4	11.0	44.0	12.7	45.6	14.4	47.2	15.1	47.2
6 Soil+12.5 mg. AmSO ₄ +1.0 g. waste	5.0	26.4	9.5	32.8	11.2	32.8	12.8	32.8	13.5	32.8
7 Soil+12.5 mg. AmSO ₄ +0.5 g. straw	4.9	33.6	7.3	47.2	7.4	44.0	—	—	—	—
8 Soil+12.5 mg. AmSO ₄ +1.0 g. straw	5.3	39.2	6.8	44.8	7.0	41.6	—	—	—	—

N in mg. : N lost expressed as mg. of AmSO₄. N as %: Percentage of total N lost on the basis of added AmSO₄ (over the corresponding controls).

TABLE V

Influence of cotton waste on the stabilisation of the nitrogen in the soil (the black cotton soil from Guntur) treated with ammonium sulphate

Treatment	2 days		6 days		12 days		16 days		20 days	
	N in mg.	N as %	N in mg.	N as %	N in mg.	N as %	N in mg.	N as %	N in mg.	N as %
1 Soil only ..	0.7	..	1.9	..	2.9	..	3.5	..	4.0	..
2 Soil+0.5 g. waste (20 tons/acre) ..	1.7	4.0	4.8	11.6	6.4	14.0	8.0	18.0	8.8	19.2
3 Soil+1.0 g. waste (40 tons/acre) ..	1.6	1.8	4.7	5.6	6.4	7.0	8.0	9.0	8.8	9.6
4 Soil+12.5 mg. AmSO ₄ (10 cwts./acre) ..	2.2	12.0	5.5	28.8	8.6	47.6	10.0	52.0	11.0	56.0
5 Soil+12.5 mg. AmSO ₄ +0.5 g. waste ..	3.1	11.2	7.6	22.4	9.1	21.6	10.7	21.6	11.5	21.6
6 Soil+12.5 mg. AmSO ₄ +1.0 g. waste ..	2.6	8.0	6.3	12.8	7.8	11.2	9.4	11.2	10.2	11.2
7 Soil+12.5 mg. AmSO ₄ +0.5 g. straw ..	3.6	12.8	4.8	15.2	4.5	13.6	—	—	—	—
8 Soil+12.5 mg. AmSO ₄ +1.0 g. straw ..	3.5	12.8	3.6	10.4	3.2	8.8	—	—	—	—

N in mg. : N lost expressed as mg. of AmSO₄. N as % : Percentage of total N lost on the basis of added AmSO₄ (over the corresponding controls).

at the rate of 20 tons per acre to the two soils; when applied at 40 tons per acre the loss was about 10 per cent. during the same period. It is unlikely that the waste itself underwent decomposition to any appreciable extent during this period. The gradual decrease in the initial loss of ammonia from the waste would suggest that a part of the nitrogen in the material might be in some labile form. It may be noted that on the basis of nitrogen content 1 g. of the waste (containing about 1 per cent. N) is equivalent to 50 mg. of ammonium sulphate.

The loss of ammonia from the soils treated with ammonium sulphate at 10 cwts. per acre was considerably reduced owing to the presence of the cotton waste. Thus from the Punjab soil the percentage loss of ammonium sulphate in 20 days was reduced from 78.4 to 47.2 and 32.8 respectively by the application of the waste material at 20 and 40 tons per acre. In the case of Guntur soil at these levels of cotton waste, the reduction was from 56 per cent. to 21.6 per cent. and to 11.2 per cent. in 20 days. A part of the nitrogen in the waste material was lost soon after its application to soil, but this loss was comparatively negligible.

These experiments show that the cotton waste material may be utilised for conserving nitrogen in soils treated with ammonium sulphate.

5. STUDIES ON THE DECOMPOSITION OF THE COTTON WASTE AND ON THE POSSIBILITIES OF EMPLOYING THE MATERIAL FOR MAKING ORGANIC MANURE

The cotton waste as such and the powdered material were used for these experiments. The extent of decomposition of the cotton material was determined by examination of the material under microscope and by observations on the micro-organisms and associated changes.

Influence of treatment of the cotton material with sulphuric acid (pre-treatment with 10 per cent. acid), other chemicals (potassium permanganate, ferrous sulphate, mixture of ferrous sulphate and lime and also ammonium sulphate), and organic wastes (raw sewage, septic sludge and actively decomposing compost materials) on its decomposition was studied. Corresponding control series with water only (tap water and distilled water) and the other organic wastes only (including straw powder only) were also maintained. Varying amounts of the cotton material, 10 g. lots to 1 lb. (the larger amounts only along with other organic wastes) were used in the experiments.

Influence of pre-treatment of the material with sulphuric acid.—Pre-treatment of the powdered material with acid was carried out as follows: the material was soaked in the dilute acid (e.g., 100 c.c. of 10 per cent. acid

for 10 g. of the material) for 20 hours and it was washed free of acid (the filtrate was orange coloured). The acid treated and washed material of cotton waste was inoculated with the aqueous extract of the original material undergoing decomposition for about 10 days (5 c.c. for 10 g. lot of the washed material) and stirred well in tap water (80 c.c. for the 10 g. lot in a shallow glass basin). Larger amounts of the material thus prepared were also mixed with compost materials. Observations extending over a month showed that pre-treatment of the material with acid had no appreciable effect on its decomposition.

Influence of soaking the material in water and also of addition of small amounts of potassium permanganate.—A few days (5 to 7 days) after keeping the cotton waste as such in water, it was observed that bacterial forms, such as *Bacillus subtilis* and *Spirillum* sp., and other micro-organisms (no fungal forms) including protozoa such as *Amæba* sp. and small ciliates developed. These organisms continued to develop; even after five weeks, large numbers of *Amæba* sp. could be seen. Such development of micro-organisms was not seen on straw powder treated with water only.

When the powdered material was treated with water (distilled water or tap water, 100 c.c. for 10 g. lots of the material in shallow glass basins), after 48 hours, a thick, fluffy scum holding gases was formed. As the scum was disturbed and gases liberated at intervals of 24 hours, the scum formation and evolution of gases took place increasingly for about 4 days. After this period the gas formation steadily decreased and at the end of 8th or 9th day it practically stopped. From the 10th day onwards it was observed that a variety of saprophytic bacteria and small forms of protozoa developed and were present throughout the period of observation, *viz.*, about 5 weeks. Similar observations were made in the case of the series in which the powdered material was treated with potassium permanganate (2 mg. of permanganate in 100 c.c. of water for 10 g. of the waste material).

When the material was suspended in water (10 g. of the powdered material in 1 litre of water) and air was bubbled through the suspension, aerobic organisms developed more rapidly and after 2 to 3 weeks, larger forms of ciliates developed; and when the aeration was stopped, in about a fortnight the latter forms of life disappeared and putrefactive changes took place.

Influence of treatment of the material with other chemicals.—Treatment of the material with ferrous sulphate or a mixture of ferrous sulphate and lime had no appreciable influence on the decomposition changes. Ammonium sulphate was effective, but not so effective as raw sewage and sludges.

Influence of treatment of the material with raw sewage.—Treatment of the material with other organic wastes, especially raw sewage and sludge, was found more effective in the disintegration of the material. Forty-eight hours after treatment of the powdered material with raw sewage (10 g. of the cotton material with 100 c.c. of detritus-free sewage in a shallow glass basin) a thin scum was found to be formed which under microscope showed as being largely due to the development of 2 or 3 species of small ciliate protozoa which were found to persist for the next 2 days. After this period, other forms of protozoa, such as *Paramecium* sp., *Vorticella* sp. and *Acineta* sp. also developed. After the 5th day, the numbers of protozoa and also of certain bacterial forms including *Spirillum* sp. steadily increased. During this period the disintegration of the cotton fibres was perceptible. After about a fortnight, the dominating organisms were a species of smaller ciliate and to a lesser extent, *Paramecium* sp., which particularly developed on the surface forming a whitish scum. One c.c. of this scum contained about 500,000 ciliates of which one-eighth to one-tenth was *Paramecium* sp. The disintegration of the cotton material by these organisms could be seen under microscope. The active development of these organisms proceeded so long as moisture was present in the medium; the material (in the dish mentioned above) became dry after 5 to 7 weeks at room temperature. That the nutrition of these organisms was mostly derived from the disintegrated cotton material was confirmed by the corresponding control series and by subsequent experiments with these organisms.

The influence of raw sewage on the destruction of the cotton fibres could be more clearly seen, when the original cotton waste (without being powdered) was used. In about 3 weeks the cotton material was found being disintegrated yielding a brown deposit and supporting a large number of ciliates, viz., *Paramecium* sp., *Euploteus* sp., *Acineta* sp., and smaller ciliates and also species of higher organisms, viz., species of Rotifer.

Influence of treatment of the material with septic sludge.—Forty-eight hours after treatment of the powdered material with septic sludge (10 g. of the cotton material, 20 g. of the sludge and 50 c.c. of water in a shallow glass basin), a thick fluffy scum due to gas formation was noticed. After the 4th day, the gas formation steadily decreased and by the 7th day gas formation practically ceased. After the 5th day, development of protozoa (*Vorticella* sp. and other ciliates) was observed in the medium, and the organisms continued to develop (the number of organisms being much less than that in the raw sewage series) so long as there was moisture in the medium. The mixture of sludge and cotton waste became dry in 3 to 5

weeks, while in the case of the corresponding control series (the sludge and water only) the material became dry in 1 to 2 weeks. The presence of cotton waste apparently served to retain moisture.

Observations on mixtures of the cotton waste and septic sludge ($\frac{1}{2}$ lb. + $\frac{1}{2}$ lb., and 1 lb. + $\frac{1}{2}$ lb. of each respectively) were also carried out. Twenty-four hours after mixing the materials and keeping the mixtures in shallow glazed pots, the volumes increased perceptibly indicating gas formation. Observations extending over 6 weeks showed that the cotton fibres were disintegrated by a variety of fungal forms including those commonly found in compost heaps.

Influence of treatment of the cotton waste with materials from compost heaps.—Treatment of the cotton waste with materials from compost heaps (10 g. of the powdered cotton waste, 20 g. of compost and 50 c.c. of water) was comparatively less effective. The micro-organisms developed to a lesser extent under these conditions.

A sample of the cotton waste was introduced into a compost heap under field conditions and allowed to undergo decomposition. The decomposing cotton material, when taken out and examined after about 7 weeks, showed the presence of fungal forms.

6. DISCUSSION

The experimental observations described above raise a few points of considerable practical importance, which may be further investigated with special reference to Indian conditions. Production of a cheap sulphonated carbon having ion exchange properties seems to be an economic and useful method of utilisation of the cotton waste, especially in areas where softening of excessively hard waters and treatment of waters containing fluorides in toxic concentrations are necessitated. At other centres, particularly in certain cotton-growing regions, the possible use of cotton waste in stabilising nitrogen in soils fertilised with ammonium sulphate may be considered. The possible utilisation of the cotton waste along with sewage and sewage sludges for preparing organic manure may also be considered.

In regard to the decomposition of the cellulosic material, it may be of interest to note the observations described in this paper as also the observations generally relating to the subject recorded by Russell (1927, 1937) and by Waksman (1931). The commonest cellulose-decomposing organisms under aerobic conditions are the actinomyces and the fungi, and it is generally assumed as the result of Waksman's work that in the soil these are more active than the bacteria, and further that they produce humus.

Rege (1927) isolated and studied certain thermophilic species of *Coprinus*, *Aspergillus* and *Acremoniella*, which decompose cellulose at high temperatures, and apparently play an important part in manure heaps. Some protozoa, e.g., those living symbiotically in termites, have been reported to take part in the decomposition of cellulose or of lignin. It is maintained that termites depend for the digestion of their food, such as woody tissue, on the protozoa present in their intestines and they are unable to feed after they are freed from protozoa by partial sterilisation (Cleveland, 1924).

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