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TITLE

Stabilization of colour of woolenized jute by radiation chemical treatment

FINAL REPORT FOR THE PERIOD

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CONTRACT NO. IAEA/BAEC-RC-1704/RB

Title of the Project:	Stabilization of colour of woolenized jute by radiation chemical treatment.
Institution where research is being carried out:	Atomic Energy Centre (Bangladesh Atomic Energy Commission).
Chief Scientific Investigator:	Dr. F.R.Al-Siddique.
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STABILIZATION OF COLOUR OF WOOLENIZED JUTE BY RADIATION CHEMICAL TREATMENT

1. INTRODUCTION

Raw jute and bleached jute when exposed to light and air becomes discoloured in a very pronounced manner to a yellowish to brownish shade. This phenomenon of discolouration is known as yellowing of jute. Yellowing is also common in woolenised jute[1].

Callow et al[2] reported that when jute is exposed to light and air all the main component undergo degradation and the lignin loses methoxy groups during irradiation with the formation of O-diphenols and ultimately coloured O-quinones. Barker[3] observed that no yellowing of jute occurs in the absence of water and the yellowing is inhibited by ethyl alcohol probably due to its dehydrating power. It is therefore expected that any treatment that would reduce the affinity of jute towards water would also reduce the rate of its discolouration.

The present project is designed to prepare jute plastic combinations that would reduce the rate of yellowing of bleached and woolenised jute through the protection of colour producing phenolic or enolic site of lignin from the action of water. Various authors attempted to reduce the colour reaction of jute either by protecting the reactive group by chemical reaction such as acetylation[2,4,5] and methylation[6] with various amount of success. Attempt has also been made to protect jute from yellowing by preventing the water molecule to come in contact with reactive site by the deposition of urea folmaldehide resin[7] into it.

In the present project various plastic was formed onto jute through radiation chemical treatment of jute in presence of acrylic monomers, in order to prevent the water molecule to come in contact with the site of colour reaction. During the course of this project variously prepared jute plastic combination (JPC), the solvent effect of the co-polymerization reaction, and water repellancy of jute has been studied.

This is the final report on the work done under the contract.

2. EXPERIMENTAL

2.1 Materials

White jute (C.capsulari) was used for these experiment, by taking the portion of jute between 50 cm to 150 cm from the bottom.

Methyl methacrylate, vonylacetate, and sodium metasilicate from BDH technical grade, Methanol, from BDH AnalaR grade, Benzene, H_2O_2, NaOH, Acetone, $NaHCO_3$, and acrylonitrile from E Merck were used.

2.2. Dewaxing of jute

The jute samples were dewaxed by soxhleting them first with benzene and then with Methanol, 8 hours each.

2.3. Bleaching of jute

The jute samples were bleached with 2 vol H_2O_2 in presence 0.1% NaOH and 0.5% $Na_2 SiO_3$, $5H_2O$ in water for 24 hours.

2.4. Weighing of jute

Jute samples were dried at 60°C in vacuum oven till constant weight according to TAPPI standard[8,9].

2.5. Purification of monomer

Ethyl acrylate, methyl methacrylate and acrylonitrile were purified by washing 4 times with 2% NaOH and then with the saturated $NaHCO_3$ soln. and finally with distilled water till alkali free. They were then distilled under vacuum. Venylacetate were purified by distilling it under vacuum.

2.6. Soaking of jute samples with monomer

The jute sample were kept soaked in the monomer solution for at least half an hour before making the soaked solution oxygen free.

2.7. Removing oxygen from monomer soaked jute sample

The monomer soaked jute samples were taken in a sample tube fitted with a ground joint socket through which it can be fitted to a stop cock, and the whole assembly (Fig.14) is then connected to the vacuum system through ground joint cone and socket. The gas from the sample were removed by repeated evacuation, freezing with liquid nitrogen, melting, evacuation, introducing oxygen free nitrogen cycle and finally the assembly removed in closed condition for irradiation.

2.8. Irradiation

Irradiation of the sample were done with a 5000 Ci γ-source at the rate of 0.1 Mrad per hour.

2.9. Removal of excess polymer

The samples after irradiation were washed with distilled water and then soxhleted with acetone for 8 hours to remove the excess polymer. The graftpolymerised jute samples were then bone dried as before and weighed.

2.10. Measurement of affinity of jute towards water

Jute samples were kept at 25°C at 65% humidity (in one experiment at 20°C and 66% humidity) for 7 days and water vapour adsorbed in terms of percent was measured by infrared ray moisture tester ULTRA X 70 manufactured by A.Gronert.

3. RESULTS AND DISCUSSIONS

3.1. Effect of γ-ray Dose on the Polymer Uptake onto Jute

Jute samples were soaked with monomer solutions in 1:1 v/v aqueous methyl alcohol and irradiated with various dose of γ-radiation. Figure: 1,2, and 3 show the amount of polymer uptake by jute due to radiation chemical reaction against the dose in Mrad from 5% Methylmethacrylate (MMA), 10% Vonyl acetate(VA), and 5% Acrylonitrile (AN) respectively.

From the results it appear that methylmethacrylate is incorporated more easily and the venyl acetate is incorporated less easily onto jute, than Acrylonitrile.

Fugure 4 shows the amount of polymer uptake by jute when it was irradiated in suspension of 10% 1:1 mixture of styrene (STY) and methylmethacrylate in 2:1 v/v methyl alcohol in water. Here the trend of polymer incorporation is almost as that in figure 1. The dose requirement for polystyrene uptake being very much higher than that for MMA uptake, it may be possible that most of the incorporated polymers here is MMA rather than STY. A further study in this respect is however necessary before a definite conclusion is made.

3.2. Effect of Concentration of Monomer on the Polymer Incorporation.

In order to study the effect of monomer concentration on the incorporation of polymer onto jute, the jute sample were suspended in various percentage of monomer solutions in 1:1 v/v aqueous methyl alcohol and irradiated with 1.5 Mrads of γ-radiation. Figures 5,6 and 7 show the amount of polymer uptake by jute due to radiation chemical reaction against the percentage of MMA, AN, and 1:1:: STY:AN respectively. From the figure it appear that the polymer incorporation increases linearly. It is also interesting to note that the mixture of AN & STY gives a higher rate of grafting than that given by AN alone. Considering the fact that the dose requirement for polystyren uptake is very much higher than that for AN uptake[12], it appears that there may be a synergistic effect in polymer incorporation when AN and STY, are present

together. Presence of this synergistic effect later confirmed by another set of experiment (Fig. 10). The same phenomenon has also been observed by Agarwal and Sreenivasan[10]. This effect was, however, not observed with MMA-STY mixture (Fig. 4).

3.3. Effect of solvent on the polymer uptake

Swelling plays an important role in the preparation of jute plastic composites. It makes the internal parts of jute fibre accessible to the monomer molecules.

In this project water and methanol was used as swelling agent. In order to determine the effect of methanol, the jute samples were suspended in 10% solution of monomers in water-methanol system having various percentage of methanol. The samples were then irradiated with 2 Mrads of γ-radiation. Figures, 8,9 and 10 shows the amount of MMA, AN, AN+STY respectively incorporated onto jute due to radiation chemical reaction against the percentage of methyl alcohol present in monomer solutions.

From Figure 8, the amount of (poly) methyl methacrylate incorporation seem to be very low at the lower concentration of methanol. Between 40 to 50% methanol concentration the uptake of MMA increses sharply, after whch it reaches a platue. Majumdar and Rapson[11] observed that for a fixed radiation dose and styrene-jute ratio, increasing amounts of water or any of the swelling agents increased the amount of polystyrene grafted to a maximum and then decrease it.

In the case of AN (Fig. 9) the methanol does not seem to have much affect whereas the incorporation of AN+STY increases continuously and sharply (Fig.10) between the methanol concentration range 45 to 90%. Here also we find that at any methanol concentration the polymer uptake is much higher when 5% AN is used in combination with 5% STY (Fig.10) than when 10% AN (Fig. 9) was used in absence of STY indicating the presence of a synerigistic effect.

3.4. Effects of Polymer Loading on the Affinity of Jute Towards Moisture

As the affinity of jute towards moisture is responsible for some of the adverse properties of jute, it was thought to be important to study the effect of polymer loading on the affinity of Jute towards moisture.

The Figure 11 shows the percentage of weight increase of (poly) MMA loaded jute due to adsorption of moisture at 25°C and 65% humidity. Adsorption of moisture by dewaxed and bleached jute and by raw jute are also been shown in this Figure. From the results it appear that the water adsorbing capacity of jute increases due to dewaxing followed by bleaching and decreases with the increase in the amount of polymer loading. At about 20% polymer loading the water adsorption capacity is reduced to 12.5% and seem to reach a platue.

The Figure 12 shows the weight increase (in percent) of jute loaded in AN and STY togather due to adsorption of moisture at 20°C and 66% humidity. From the results it appear that the water adsorbing capacity of jute decreases with the increase in the amount of polymer loading. Unlike the water absorbing capacity of poly-MMA incorporated jute, the decrease in the water adsorption capacity of poly-(AN+STY) incorporated jute per unit polymer loading seem to increase with the increase in polymer loading.

3.5. Effect of Swelling Agent During Polymer Incorporation on the Affinity of Jute Towards Moisture

Dewaxed jute samples were irradiated with 0.8 Mrad (dose requirement for the polymerisation of ethyl acrylate is 0.5 to 0.7 Mrad[12]) of γ-ray in 2% (maximum amount that could be dissolved at room temp.) ethyl acrylate (EA) solution in water in the presence of various concentrations of formic acid.

The effect of formic acid on the incorporation of poly-EA onto jute are shown in Table 1. The Figure 13 shows the effect of concentration of formic acid used during poly-EA incorporation on the moisture adsorption of jute at 25°C and 65% humidity. From the results in Table-1 it is rather difficult to see much of an influence of the concentration of formic acid on the amount of polymer uptake by the jute after an initial increase when 0.5% formic acid concentration was used. However, from Figure-13, it appears that the affinity of jute poly-EA composite towards

Table-1

EFFECT OF FORMIC ACID ON THE ETHYL ACRYLATE INCORPORATION

Concentrations of the ethyl acrylate(per cent)	Concentration of formic acid (percent)	Increase in weight of jute due to ethyl acrylate incorporation(per cent)
2	0.0	1.40*
2	0.5	2.18
2	1.0	2.02
2	2.0	2.00
2	3.0	1.37
2	4.0	1.75
2	5.0	1.70

*Mean of 9 irradiated samples.

water vapour decrease with the increase in concentration of formic acid. The poly-EA seems to have blocked the hydrophillic groups of jute more efficiently with increasing amount of swelling agent like formic acid. Better swelling of jute by formic acid might have rendered the polymer to form more intimately inside jute and there by blocked the hydrophillic group more efficiently.

4. PAPER PUBLISHED ON WORK DONE UNDER THE CONTRACT

Al-Siddique, F.R., Zaman, M.A. Effect of formic acid on the radiation induced graft copolymerisation of ethylacrylate onto jute and the affinity of the copolymer towards moisture, Nucl.Sc. & Appl., 9B, 80-82 (1976).

Al-Siddique, F.R. et al., Modification of jute fibre by graft co-polymerisation, presented to 2nd Annual Bangladesh Sc. Conf. 1977 Abstract no. Chem. 22.

Al-Siddique, F.R. et al., Modification of jute fibre by graft co-polymerization, Report of Atomic Energy Centre,Dacca. AECD/CH/21, (1978).

5. REFERENCES

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3. Sarkar, P.B.,Current Sci., 16, 287(1947).

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7. Callow, H.J., J.Text.Inst., 41, T404 (1950).

8. TAPPI Standard Method T210 m-53.

9. Pulp and Paper Research Institute of Canada, Standard Testing Procedure No.C-M-1.

10. Agarwal, S.R. and Srinivasan, A., Indian J.Technol., 12, 456-59 (1974).

11. Majumdar, S.K. and Rapson, W.H., Text. Res.J., 34 1007-15 (1964).

12. Hills and McGahan, D.J., ATOM, No. 176, June, 1971.

6. EXPLANATION OF DEPARTURE FROM THE LEVEL OF ACTIVITY FORESEEN BY THE CONTRACT

Effect of polymer loading on the rate of yellowing of jute could not be studied due to repeated failure of the Colour Difference Meter. Now we are waiting for a supply of some spare parts ordered to Hunter Lab. Inc. We are expecting to start work on the above study as soon as the spares arrive and the Colour Difference Meter comes to the working condition.

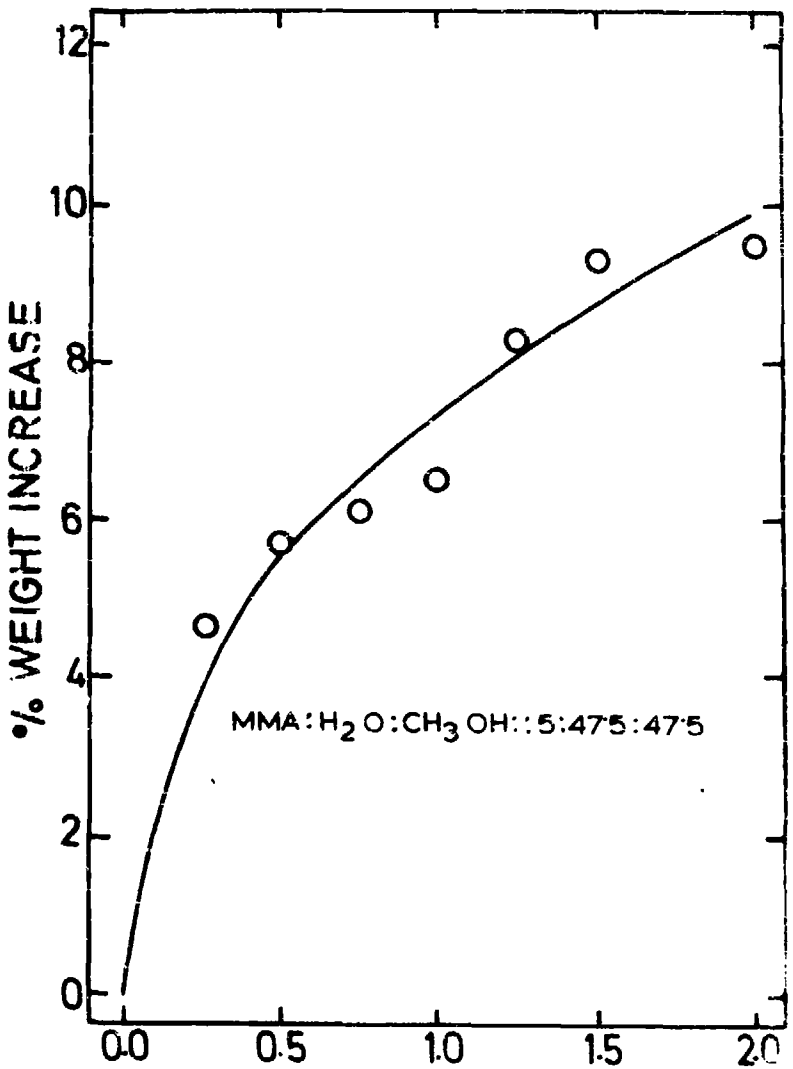

% WEIGHT INCREASE
12
10
8
6
4
2
0
0.0
0.5
1.0
1.5
2.0
MMA:H2O:CH3OH::5:47·5:47·5
TOTAL DOSE IN MRADS

FIG.1.

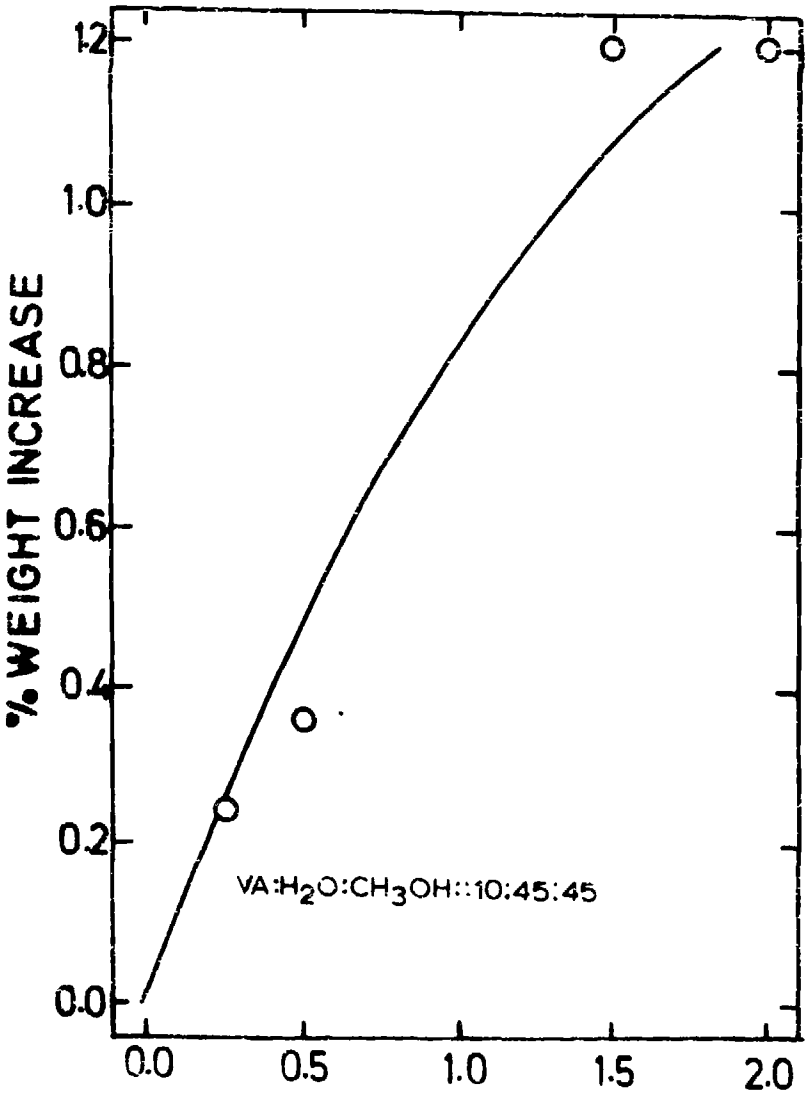

% WEIGHT INCREASE
1.2
1.0
0.8
0.6
0.4
0.2
0.0
0.0
0.5
1.0
1.5
2.0
VA:H2O:CH3OH::10:45:45
TOTAL DOSE IN MRADS

FIG.2.

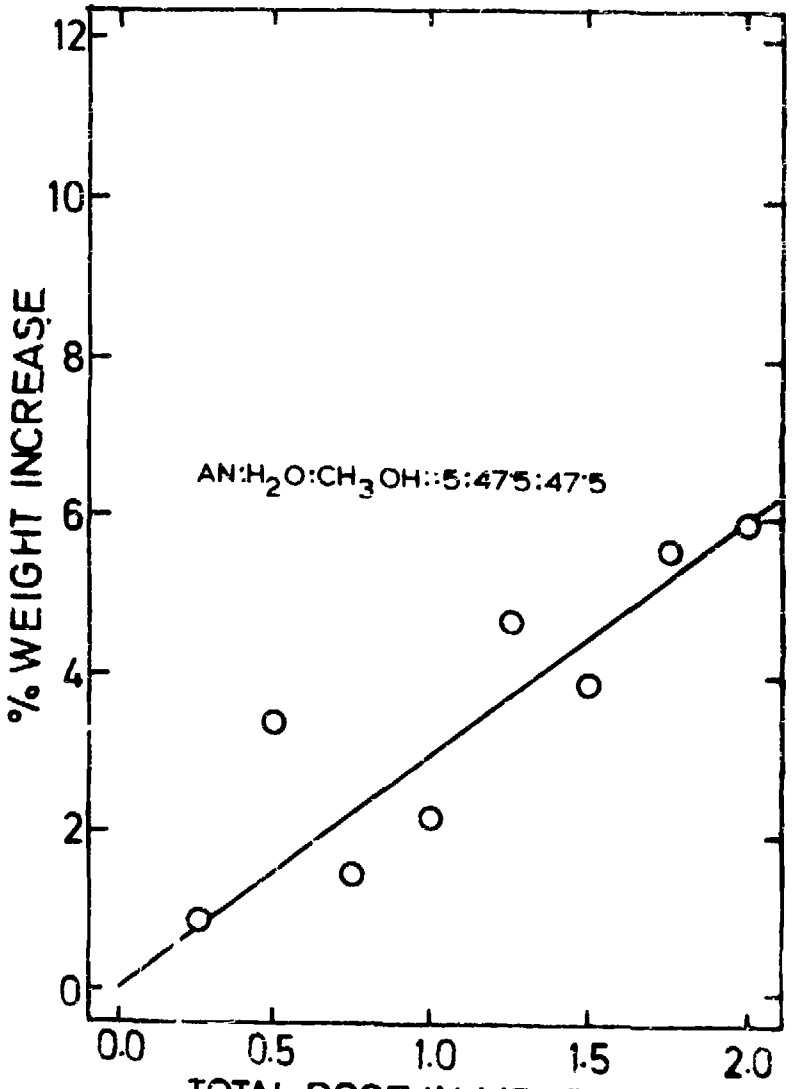

% WEIGHT INCREASE
AN:H2O:CH3OH::5:47·5:47·5
TOTAL DOSE IN MRADS
0.0
0.5
1.0
1.5
2.0
0
2
4
6
8
10
12

FIG.3.

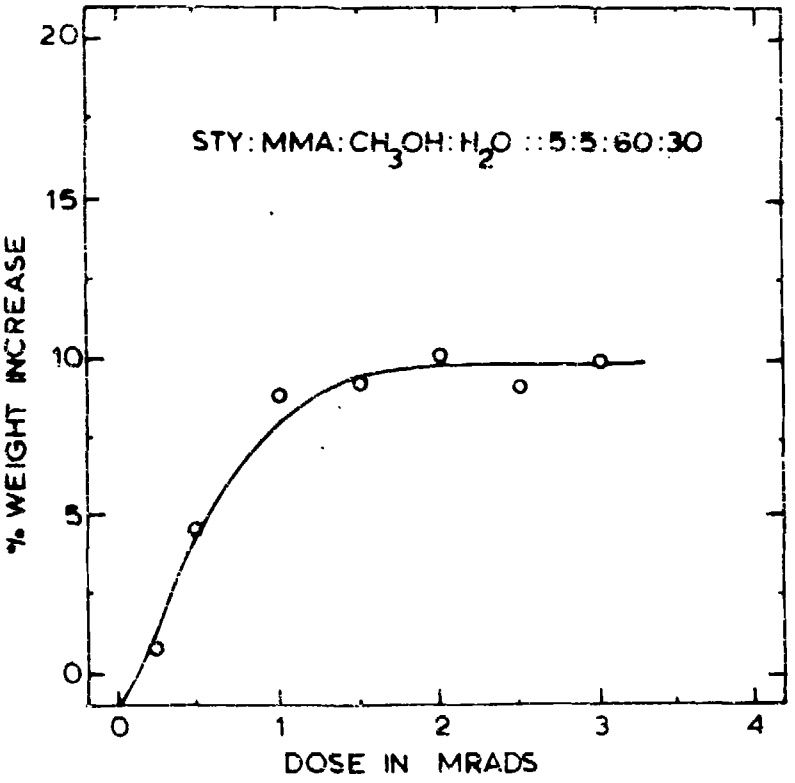

% WEIGHT INCREASE
STY:MMA:CH3OH:H2O ::5:5:60:30
DOSE IN MRADS
0
1
2
3
4
0
5
10
15
20

FIG.4

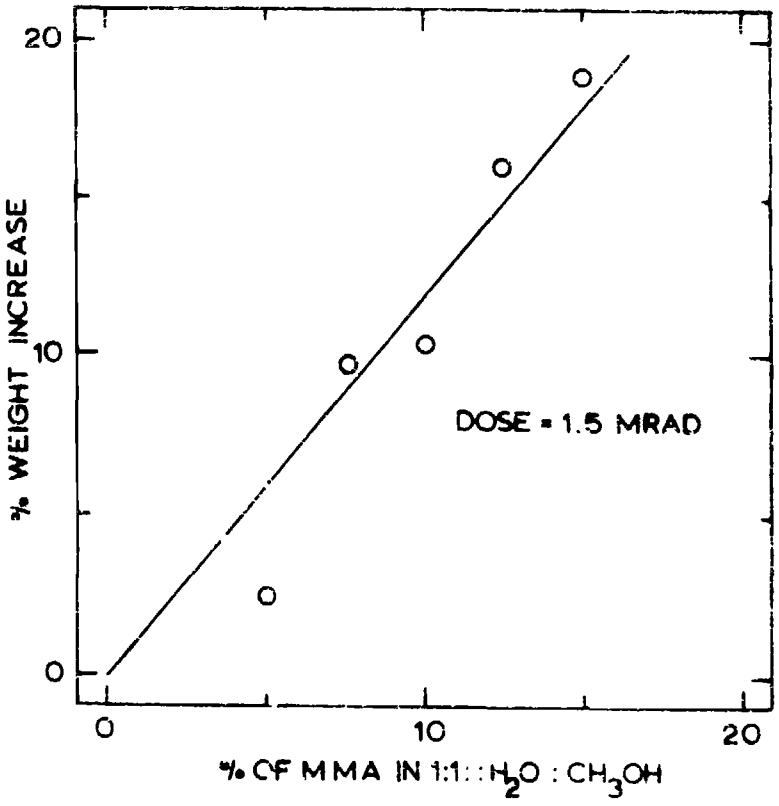

20
10
0
% WEIGHT INCREASE
DOSE = 1.5 MRAD
0
10
20
% OF MMA IN 1:1:: H_2O : CH_3OH

FIG.5

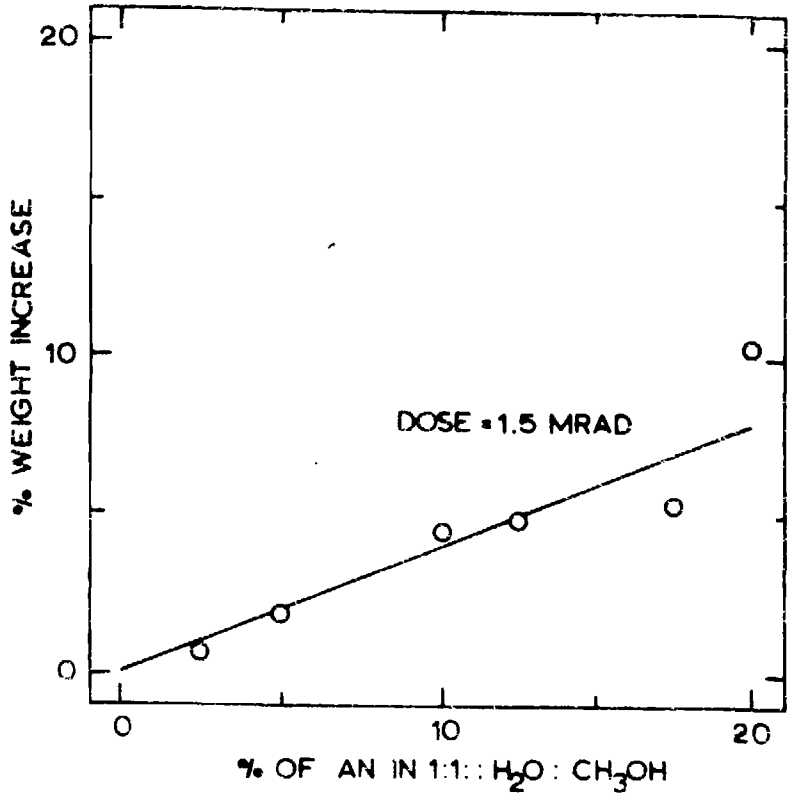

20
10
0
% WEIGHT INCREASE
DOSE = 1.5 MRAD
0
10
20
% OF AN IN 1:1:: H_2O : CH_3OH

FIG.6

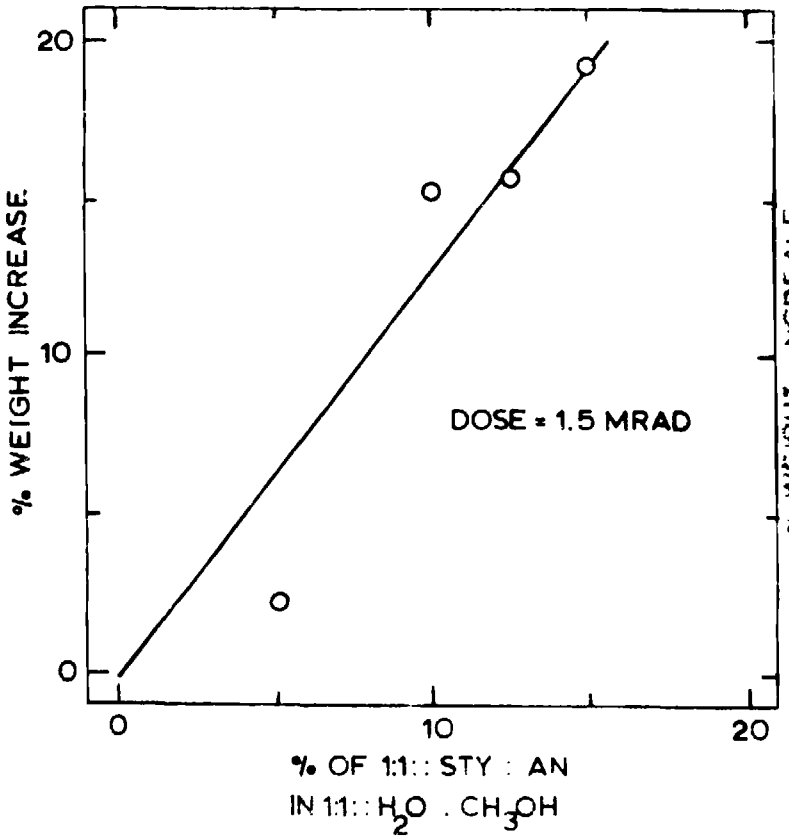

% WEIGHT INCREASE
20
10
0
0
10
20
DOSE = 1.5 MRAD
% OF 1:1:: STY : AN
IN 1:1:: H_2O . CH_3OH

FIG. 7

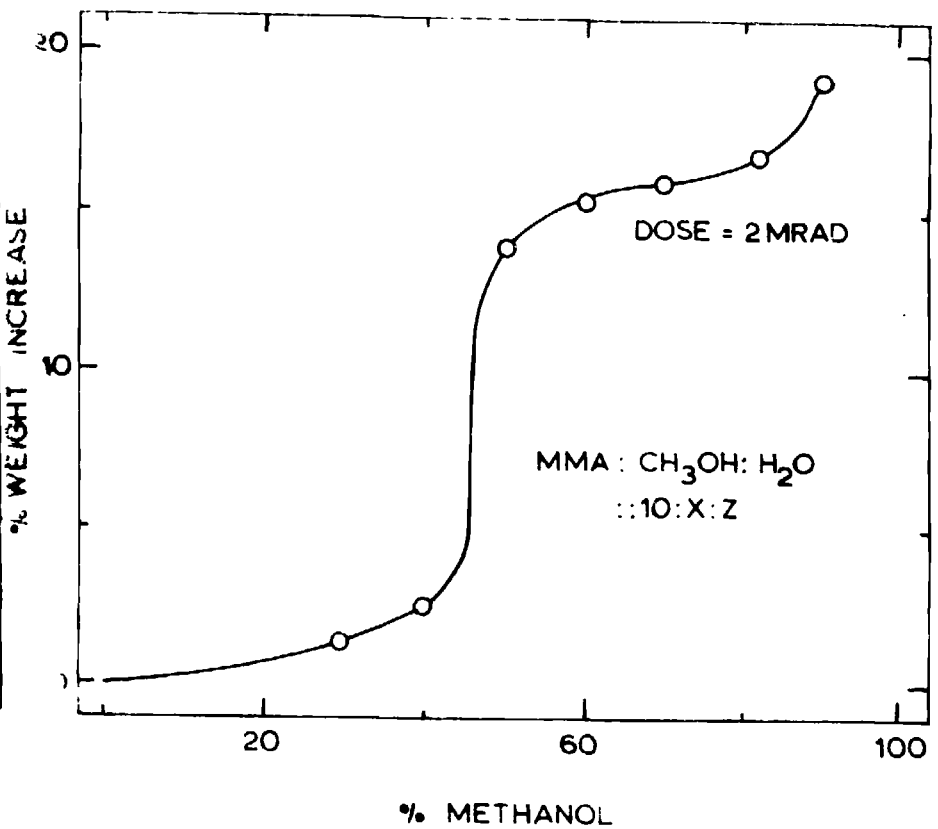

% WEIGHT INCREASE
20
10
20
60
100
DOSE = 2 MRAD
MMA : CH_3OH: H_2O
::10:X:Z
% METHANOL

FIG. 8

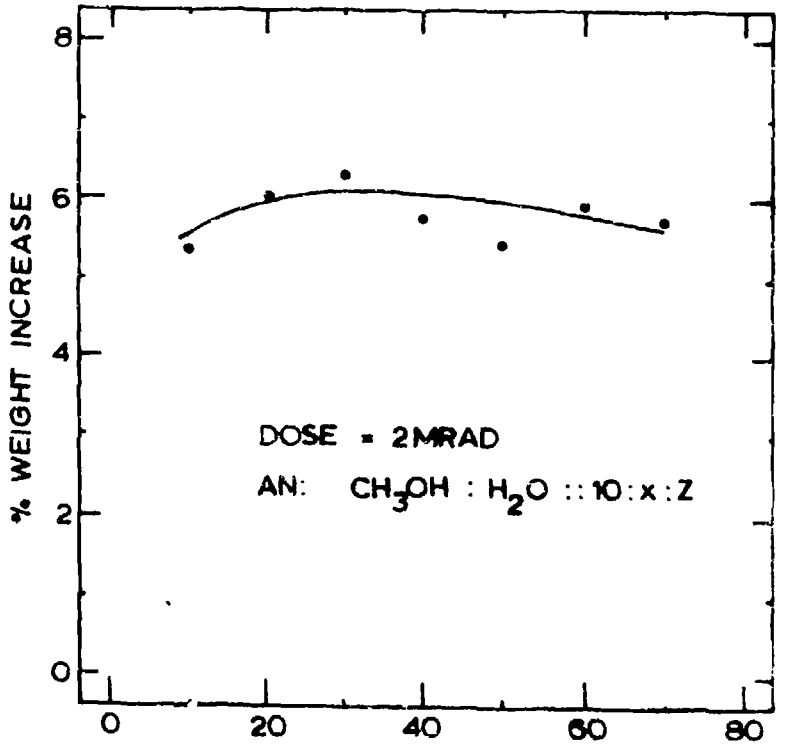

% WEIGHT INCREASE
8
6
4
2
0
0
20
40
60
80
DOSE = 2MRAD
AN: CH3OH : H2O :: 10 : x : Z
% METHANOL

FIG. 9

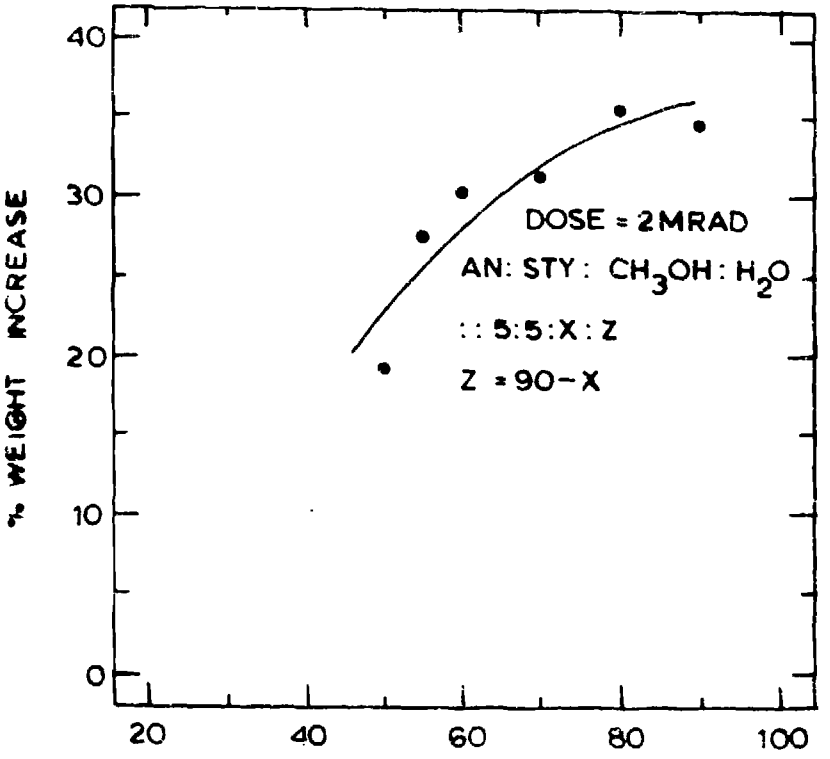

% WEIGHT INCREASE
40
30
20
10
0
20
40
60
80
100
DOSE = 2MRAD
AN: STY : CH3OH : H2O
:: 5 : 5 : X : Z
Z = 90 − X
% METHANOL

FIG. 10

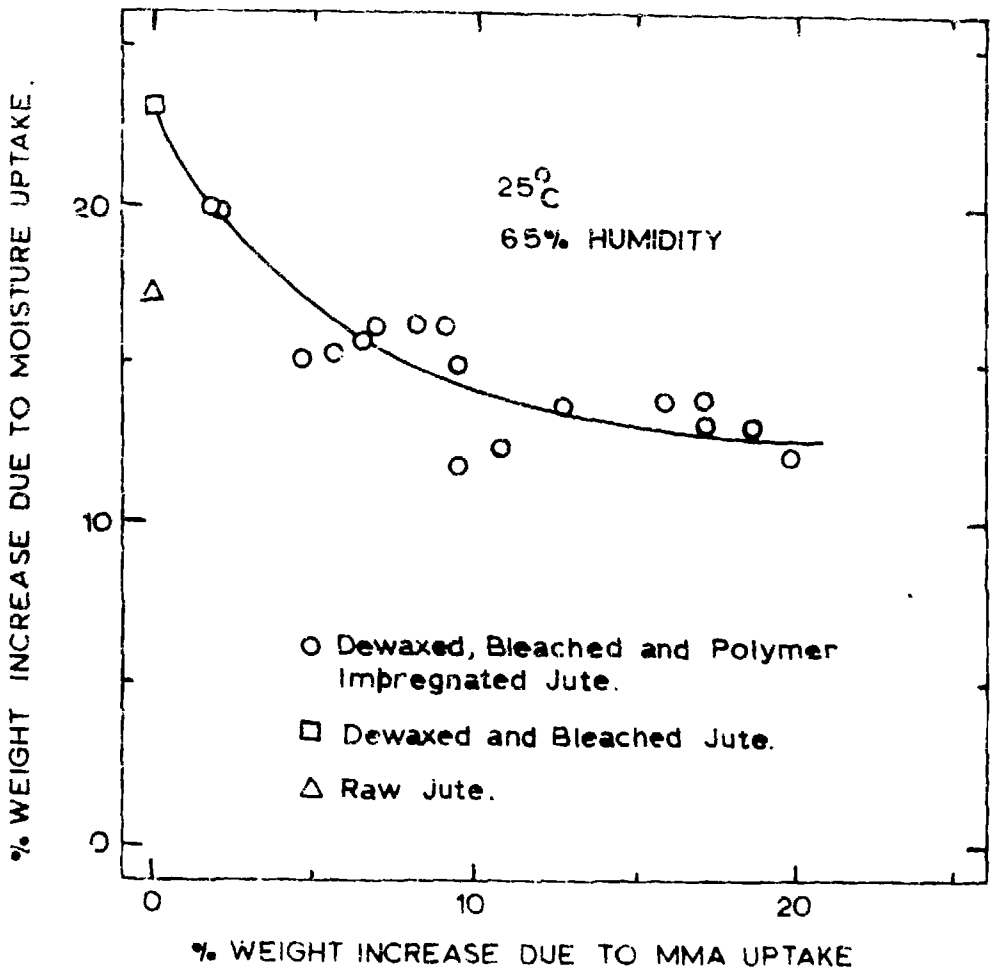

% WEIGHT INCREASE DUE TO MOISTURE UPTAKE.
20
10
0
25°C
65% HUMIDITY
O Dewaxed, Bleached and Polymer Impregnated Jute.
□ Dewaxed and Bleached Jute.
△ Raw Jute.
0
10
20
% WEIGHT INCREASE DUE TO MMA UPTAKE

FIG 11

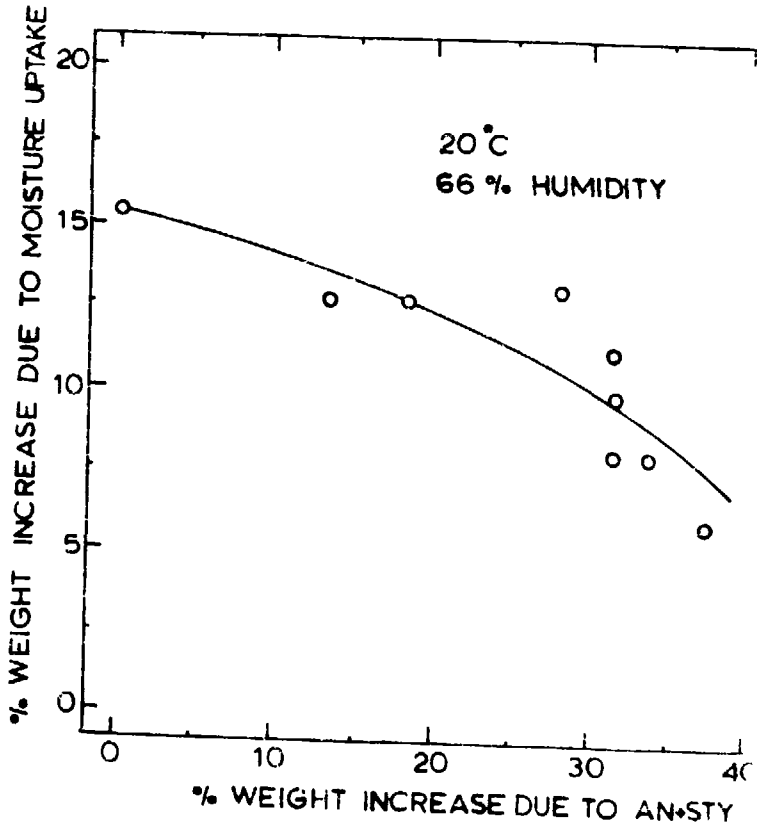


FIG. 12

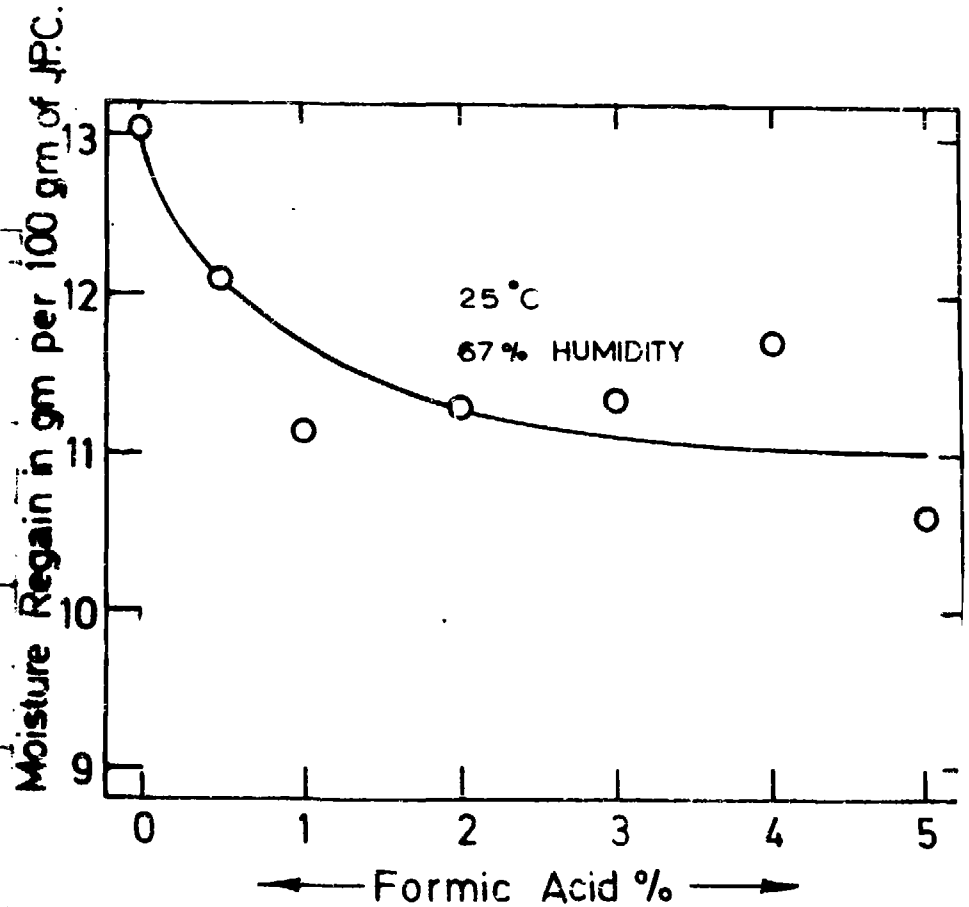
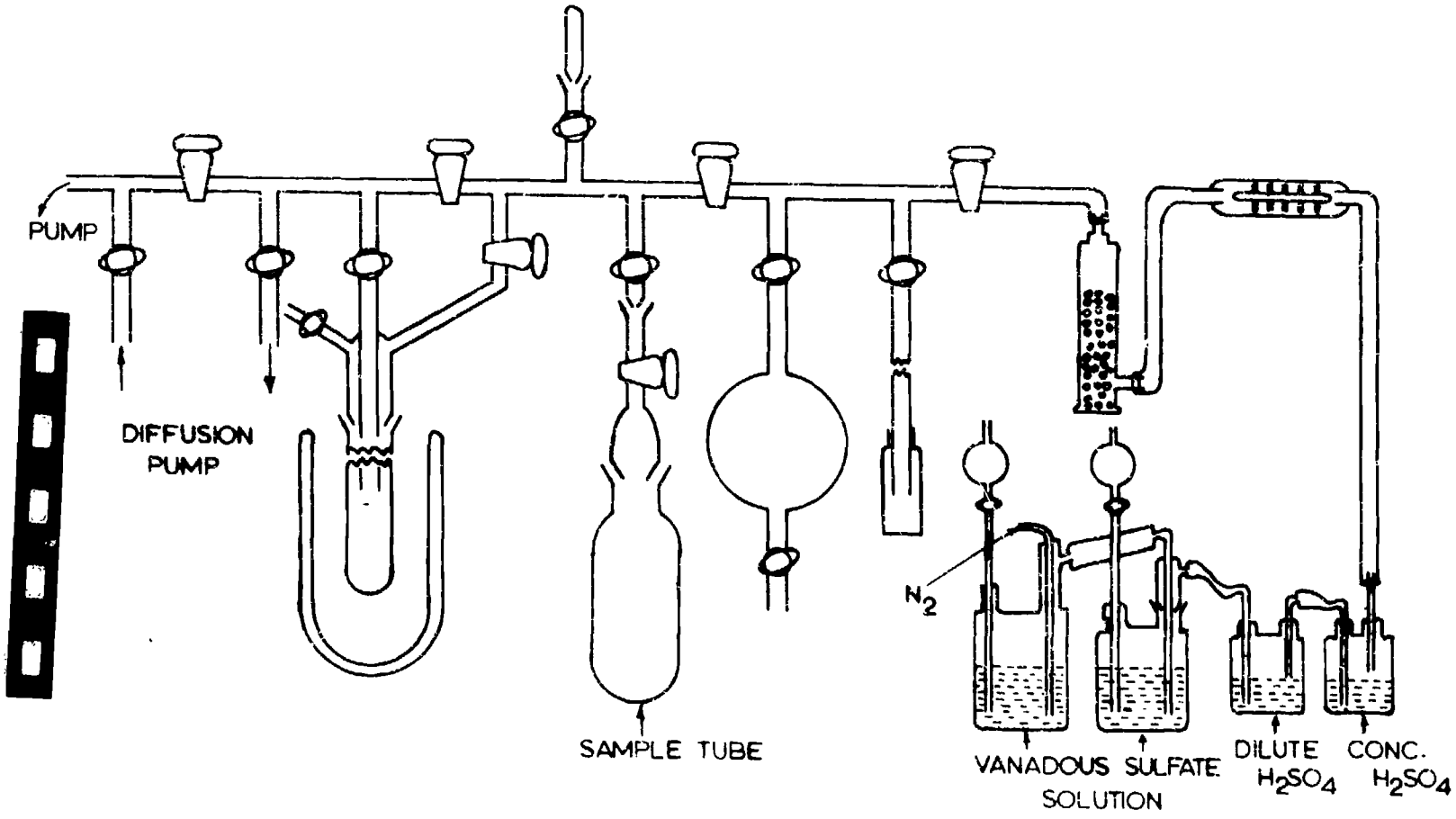


FIGURE. 13



VACUUM SYSTEM FOR REMOVAL OF O_2 FROM THE SAMPLE

FIGURE 14