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DEPARTMENT OF SCIENTIFIC AND INDUSTRIAL RESEARCH AND FIRE OFFICES' COMMITTEE
JOINT FIRE RESEARCH ORGANIZATION

THE EFFECT OF LAURYL PENTACHLORPHENOL TREATMENT ON THE RESISTANCE OF UNLINED CANVAS FIRE HOSE TO ATTACK BY FUNGI

by

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Summary

An investigation has been made to determine the effect of lauryl pentachlorphenol on the resistance to mildewing of unlined canvas fire hose. The tests were performed on hose obtained from four manufacturers.

The treatment had no detrimental effect on the bursting strength or rate of percolation of the hose.

Resistance of treated hose to attack by Chaetomium globosum and Aspergillus niger showed a marked improvement over untreated hose.

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Report of Special Investigation for the Ministry of Works.

Introduction

Various tests on unlined canvas fire hose treated with sodium pentachlorphenate were carried out in 1945 (1) and it was shown from soil burial tests that the treatment improved the mildew resistance of the hose.

In September, 1952, the Ministry of Works asked the Joint Fire Research Organization to determine the effect of lauryl pentachlorphenol treatment on resistance to mildew and the serviceability of fire hose. Previous tests had shown burnettizing to give no protection against Chaetomium globosum (2). The hose for the tests was supplied by four manufacturers each of whom supplied one treated and one untreated sample. Details of the hose are given in Table 1.

Methods of testing

(a) Bursting pressure

Three 3 ft. long specimens of each type of hose were attached in turn to a pressure head connected to a hydraulic accumulator. Each specimen was allowed to soak at a pressure of 100 lb./sq.in. for 30 minutes and the pressure was then increased at the rate of 100 lb./sq.in. per minute until failure occurred. The results of these tests are given in Table 2.

(b) Percolation

For the percolation tests three specimens, each 3 ft. long, of each type of hose were conditioned in an atmosphere of relative humidity 60 per cent and temperature 105°F until their weights remained constant over a period of 24 hours. The specimens were then filled with water and the pressure was gradually increased to a value of 100 lb./sq.in. over a period of 2 minutes. The quantity of water which had percolated through the hose was measured at the ends of further periods of 5, 10, 15, 20, 30 & 45 minutes, the pressure being maintained at 100 lb./sq.in. throughout the test. The results of these tests are given in Table 3.

(c) Mildew resistance

(i) Chaetomium globosum

The resistance to mildew attack of each type of hose was assessed from the loss of tensile strength of warp specimens exposed for various periods to a culture of Chaetomium globosum. Preparation of the culture and exposure of the specimens were carried out by the Mycology Section of the Forest Products Research Laboratory of the Department of Scientific and Industrial Research and the method used was based upon that described by Abrams (3).

The warp tensile strength was determined by cutting 1 inch wide specimens from each type of hose and producing strain over a six inch length at the rate of $1\frac{1}{2}$ inches per minute. The tests were performed on wet specimens which had been soaked in water for 24 hours.

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A number of the strips to be used for tensile strength tests were leached for 6 hours in an aspirator bottle in which the water was changed completely approximately four times per minute. After drying, both leached and unleached specimens were exposed to fungal attack for periods varying from 2 to 16 days, the ends of the specimens being coated with wax to restrict the attack to the central portion.

The specimens were sprayed with a suspension of spores from an atomizer spray and placed so that the central portions made contact with a culture of spores growing on 2 inch squares of filter paper. This culture had been allowed to grow for 3 days at 30°C in a carbohydrate-free inorganic agar of pH 6·8. All specimens were dried after the appropriate incubation period and later soaked in water for the 24 hours prior to the strength tests. The mean values of the tensile strengths of mildewed and un-mildewed hose are given in Tables 4A, 4B, 4C and 4D. The average of results for treated and untreated hose is shown graphically in Figures 1-4.

(ii) Aspergillus niger

Without prior sterilisation, three 2 inch squares of hose of each type were sprayed with a spore suspension of *Aspergillus niger* and were firmly pressed down on to petri dish cultures of this fungus, on 2 per cent malt agar. The cultures, which were three days old, were incubated at 25°C for 32 days. The condition of some of the samples after incubation is shown in Plate 1 and the makes are identified in Table 5.

(d) Lauryl pentachlorphenol content

The estimation of the lauryl pentachlorphenol content of the hose was carried out by the Government Chemists Department of the Ministry of Works, the method used is described in Appendix I. The results of the estimation are given in Table 6.

Discussion

The results of the bursting pressure, percolation, and mildew resistance tests have been analysed statistically. For the final percolation and the strength of the hose these analyses were based on the logarithms of the results, and the results of the various tests are discussed below.

(a) Bursting pressure

Statistical analysis shows that for the three flat woven hoses there was no consistent change in the bursting strength due to the rot-proofing treatment. For the three samples of one make of circular woven hose however rot-proofing treatment increased the bursting strength by an average of 90 lb./sq.in.

The results in Tables 4A, 4B, 4C and 4D show that the initial strength of treated hose was somewhat lower than that of untreated hose. This however does not appear to have affected the bursting strength.

(b) Percolation

The measurements of percolation showed the customary large variation between samples of the same make of hose and a statistical analysis of the final rates of percolation showed that there was no significant difference due to the treatment or between the four makes of hose.

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(c) Mildew resistance

(i) Chaetomium globosum

The four makes of hose behaved similarly up to 8 days of incubation. A statistical analysis of the results (see Appendix II) showed that the leaching had no effect on the mildew resistance of the hose. A feature of interest shown by the statistical analysis is that, as in the previous tests [2], the second and third order interactions, when pooled together gave a variance which was significantly greater than the variation between three specimens from the same length. It was for this reason that the number of specimens tested for each condition was reduced from 6 to 3 thus halving the labour involved in the tests. It is possible in future tests that a further economy on these or other lines can be achieved.

Figures 1, 2, 3 and 4 show the following features of interest:

The lauryl pentachlorphenol treatment reduces the rate of deterioration of the hose when attacked by Chaetomium globosum, by a factor of over 4 (see Table 7); the rate of deterioration being defined as $-\frac{1}{s}\frac{ds}{dt}$, where "s" is the strength at any time.

The rates of deterioration appear to be approximately proportional to the strength remaining at any time: complete proportionality would be in accordance with the law

$$-\frac{ds}{dt} = ks$$

so that $\log_e$"s" would vary linearly with time.

This is seen to be so except in two instances. The first is at short periods of incubation where the rates of deterioration of treated and untreated hose are similar. This initial deviation from the exponential law is probably due to an initial effect in mildewing treatment. The second occurs after 16 days incubation in the case of makes A and D where the strength after 16 days incubation is less than that given by the exponential law. It is of interest that in makes B and C the content of lauryl pentachlorphenol was only a little below 1 per cent but in makes A and D it was 0·55 per cent and 0·35 per cent respectively and the reduction in strength was greater for the hose with the low pentachlorphenol content. This suggests that the rate of deterioration increased after a time dependent on the lauryl pentachlorphenol content. Until the strength of the hose was reduced to approximately one-third of its initial value the percentage content of lauryl pentachlorphenol does not appear to have been critical against Chaetomium globosum providing it is over 0·35 per cent. This might then be regarded as the minimum effective concentration.

(ii) Aspergillus niger

The condition of each group of three samples was uniform after incubation. All untreated samples were heavily overgrown by the test fungus. None of the preservative treated samples showed any fungal growth and all had inhibited growth of the fungus on the surrounding agar to some extent; this was most marked in the case of leached samples of hose BLT.

Very moist areas on the treated samples show as dark patches in Plate 1.

Conclusions

(a) There is some evidence that the pentachlorphenol reduces the tensile strength of the hose fabric but there is no evidence that the bursting strength of the flat woven hose is affected. On the other hand the bursting strength of the one sample of circular woven hose was increased.

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(b) The percolation was not observed to be affected by the treatment.

(c) The 6 hour period of leaching had no effect on the mildew resistance of the hose.

(d) (i) The resistance of hose to attack by Chaetomium globosum is improved when treated with lauryl pentachlorphenol, a content of only 0·35 per cent of this chemical being sufficient to delay rapid deterioration until the strength of the hose was reduced to approximately one-third of its initial value.

(d) (ii) Lauryl pentachlorphenol treatment markedly reduces the growth of 'Aspergillus niger'.

Acknowledgments

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References

(1) A. Bailey and P. Wright. "Investigation of a method of rot-proofing canvas delivery hose". Department of Scientific and Industrial Research and Fire Offices' Committee Joint Fire Research Organization. F.P.E. Note 16/1949.

(2) J. F. Fry, J. A. Gordon and P. H. Thomas. "The effect of burnettizing, alkali-soluble content and specification on some properties of unlined canvas fire hose". Joint Fire Research Organization. F.R. Note 49/1953.

(3) E. Abrams. "Microbiological deterioration of organic materials - its prevention and methods of test". United States Department of Commerce National Bureau of Standards. Publ. No. 188, 1948.

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TABLE 1

Details of hose

Make	Type of weave	Remarks
A - [redacted]	Flat	Rot-proofing treatment on hose
B -	Flat	Rot-proofing treatment on yarn
C -	Flat	" " " " "
D -	Circular	" " " " "

TABLE 2

Bursting pressure - lb./sq. in.

Make	Condition	Mean of three tests
A	Treated Untreated	440 465
B	Treated Untreated	365 415
C	Treated Untreated	415 385
D	Treated Untreated	715 625
σ on mean value = 20		

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TABLE 3

Initial and final percolation - gal./5 min.

Make A				Make B				Make C				Make D			
Treated		Untreated		Treated		Untreated		Treated		Untreated		Treated		Untreated	
Initial	Final	Initial	Final	Initial	Final	Initial	Final	Initial	Final	Initial	Final	Initial	Final	Initial	Final
0.60	0.27	2.90	0.95	1.00	0.09	0.80	0.27	1.08	0.11	1.40	0.10	0.73	0.13	2.61	0.24
0.55	0.18	0.73	0.20	3.80	0.38	1.73	0.15	1.36	0.15	3.25	0.30	0.55	0.13	1.51	0.30
1.28	0.26	0.79	0.20	3.85	0.47	1.90	0.19	1.33	0.13	1.15	0.10	1.68	0.22	5.45	1.49

The initial rate is derived from the percolation from 0 to 5 minutes and the final rate from the mean percolation from the 31st to the 45th minute.

TABLE 4A

Warp tensile strength after various periods of incubation - lb. per in.

Make A

Period of incubation (days)	Treated		Untreated	
	Leached	Unleached	Leached	Unleached
0	823	885	927	963
2	---	---	822	830
4	695	862	365	432
8	584	498	55	106
16	245	91	---	---

TABLE 4B

Warp tensile strength after various periods of incubation - lb. per in.

Make B

Period of incubation (days)	Treated		Untreated	
	Leached	Unleached	Leached	Unleached
0	727	723	855	905
2	---	---	835	772
4	542	567	334	458
8	506	320	83	120
16	337	310	---	---

TABLE 4C

Warp tensile strength after various periods of incubation - lb. per in.

Make C

Period of incubation (days)	Treated		Untreated	
	Leached	Unleached	Leached	Unleached
0	758	770	925	892
2	---	---	536	546
4	756	616	368	296
8	450	429	65	26
16	278	370	---	---

TABLE 4D

Warp tensile strength after various periods of incubation - lb. per in.

Make D

Period of incubation (days)	Treated		Untreated	
	Leached	Unleached	Leached	Unleached
0	798	790	905	840
2	---	---	812	594
4	527	585	321	397
8	421	486	31	104
16	25	158	---	---

TABLE 5

Identification of makes

Make	Condition	Ref. No. on Plate 1
A	Treated Untreated	BLP BLO
B	Treated Untreated	BLR BLQ
C	Treated Untreated	BLT BLS
D	Treated Untreated	BLV BLU

TABLE 6

Lauryl pentachlorphenol content of treated hoses

Make	L.P.C.P. content %
A	0.55
B	0.95
C	0.8
D	0.35

TABLE 7

Rate of exponential deterioration of hose

This is defined as $k = -\frac{1}{S}\frac{dS}{dt}$ and is calculated from the slope of the straight lines in Figures 1-4.

Make	Condition of hose	$(day)^{-1}$
A	Treated	0.08
	Untreated	0.40
B	Treated	0.07
	Untreated	0.35
C	Treated	0.08
	Untreated	0.37
D	Treated	0.09
	Untreated	0.35

APPENDIX I

ESTIMATION OF THE LAURYL PENTACHLORPHENOL CONTENT OF CANVAS HOSE

Reagents

1. 4 Normal caustic soda.
2. Hydrochloric acid (concentrated).
3. N/10 Hydrochloric acid.
4. Chloroform (pure).
5. Copper-pyridine reagent.
   Dissolve 3.0g pure copper sulphate ($CuSO_4 . 5 H_2O$) in 60 ml water. To 6 ml of this solution add immediately before use 4 ml pure pyridine. It is essential that the pyridine should be of the highest quality.
6. Primary standard
   Dissolve 0.6g Pentachlorphenol (recrystallised from benzene) and 0.4g lauric acid in about 100 ml water and 8 ml N. caustic soda. Make up to 1 litre with water. This solution is stable for many months if stored in a well-stoppered bottle in the dark.

Method

Weigh out 5.0g of canvas hose cut in pieces about ¼ inch square and place them in a 1 litre round bottom glass flask fitted by means of a ground glass joint with a Liebig type condenser. Add to the flask 60 ml 4N caustic soda and boil under reflux for 2 hours. Wash down the condenser into the flask with a little water and add further water to a total of 150 ml, followed by 75 ml hydrochloric acid (conc). By means of a connector with ground glass joints turn the condenser into a position for distillation and distil until at least 200 ml of distillate have been collected. Transfer the distillate to a 500 ml separating funnel. Wash the condenser and receiving flask with chloroform and transfer the washings to the separating funnel, using a total of 40 ml chloroform. Shake well and allow to separate. Run off the chloroform layer into a 100 ml separating funnel and add 10 ml of the copper-pyridine reagent. Shake and allow to separate. Run off the chloroform layer through a filter paper into a 50 ml calibrated flask. Wash the copper-pyridine reagent with a further 5 ml of chloroform and run through the filter paper into the 50 ml flask. Bulk to the mark with chloroform.

Compare the colour of the chloroform solution with that produced by a known quantity of the standard solution treated as follows:-

Take a known quantity of standard solution, add one tenth the volume of N/10 Hydrochloric acid and bulk to 250 ml with water. Extract with 40 ml chloroform and separate. Add 10 ml of copper-pyridine reagent to the chloroform layer, shake and separate. Run off the chloroform layer through a filter paper into a 50 ml calibrated flask. Wash the copper-pyridine reagent with a further 5 ml chloroform and run through the filter paper into the 50 ml flask. Bulk to the mark with chloroform.

50 ml of primary standard solution is equivalent to 1 per cent Lauryl Pentachlorphenol on the sample of canvas hose.

A blank determination on the reagents should be carried out and due allowance made for any colour produced.

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APPENDIX II

STATISTICAL ANALYSIS OF STRENGTH AFTER INCUBATION PERIODS OF 0, 4 AND 8 DAYS

ANALYSIS UNDERTAKEN ON LOG_{10} (STRENGTH IN lb/in.)

Factor	Sum of squares	Degrees of freedom	Mean squares
T rot-proofing treatment	4.137	1	4.14
L Leaching	0.011	1	0.011
I Incubation period	12.177	2	6.09
M Makes	0.185	3	0.062
TxL	0.035	1	0.035
TxI	5.040	2	2.52
TxM	0.201	3	0.067
LxI	0.006	2	0.003
LxM	0.153	3	0.051
IxM	0.171	6	0.029
TxLxI	0.048	2	0.024
IxLxM	0.105	6	0.018
TxIxM	0.156	6	0.026
LxTxM	0.101	3	0.034
TxLxIxM	0.135	6	0.023
Variation between specimens	1.201	96	0.0125

None of the second order interactions are significant compared with the third order interaction and the pooling of all these gives a variance of 0.024 with 23 degrees of freedom which is significantly greater than the variation between specimens

This value was therefore used to estimate the significance of the other variances.

Of the first order interactions other than the "Treatment x Incubation" interaction only the "treatment x makes" and "leaching x makes" interactions are large although their significance does not reach the 5 per cent level. The "Treatment x Leaching", "Leaching x incubation", and "incubation x makes" interactions can be pooled with the residual to give a residual of 0.024 with 32 degrees of freedom and the significance level of the "leaching x makes" interaction is then found to be approximately 12 per cent. Subsidiary analyses for the separate makes of hose do not, however, show any marked effect due to leaching. The large magnitude of the "treatment x makes" interaction which has a significance level of approximately 7 per cent might mean that the difference between treated and untreated hose varies significantly between the four makes of hose. An inspection of the results however does not indicate that this difference is correlated with the differences in pentachlorphenol content or with the method of applying the treatment i.e. yarn treated and hose treated fabric.

The fact that the main "Treatment" effect is not significantly larger than the significant "treatment x incubation" interaction is due to the treated hose being weaker before mildewing but stronger after 4 and 8 days mildewing.

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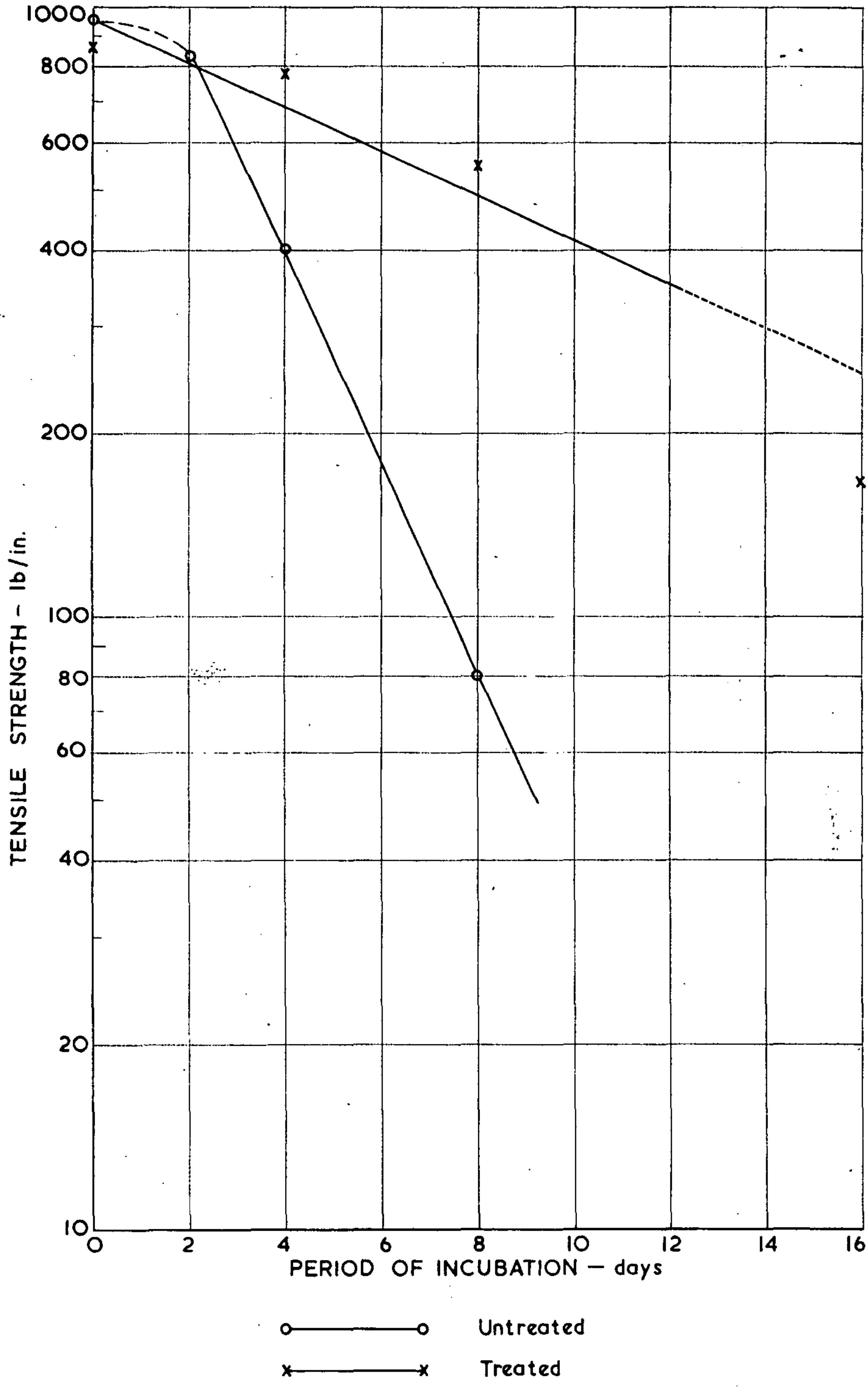

FIG. 1. THE VARIATION OF TENSILE STRENGTH WITH PERIOD OF INCUBATION. MAKE A.

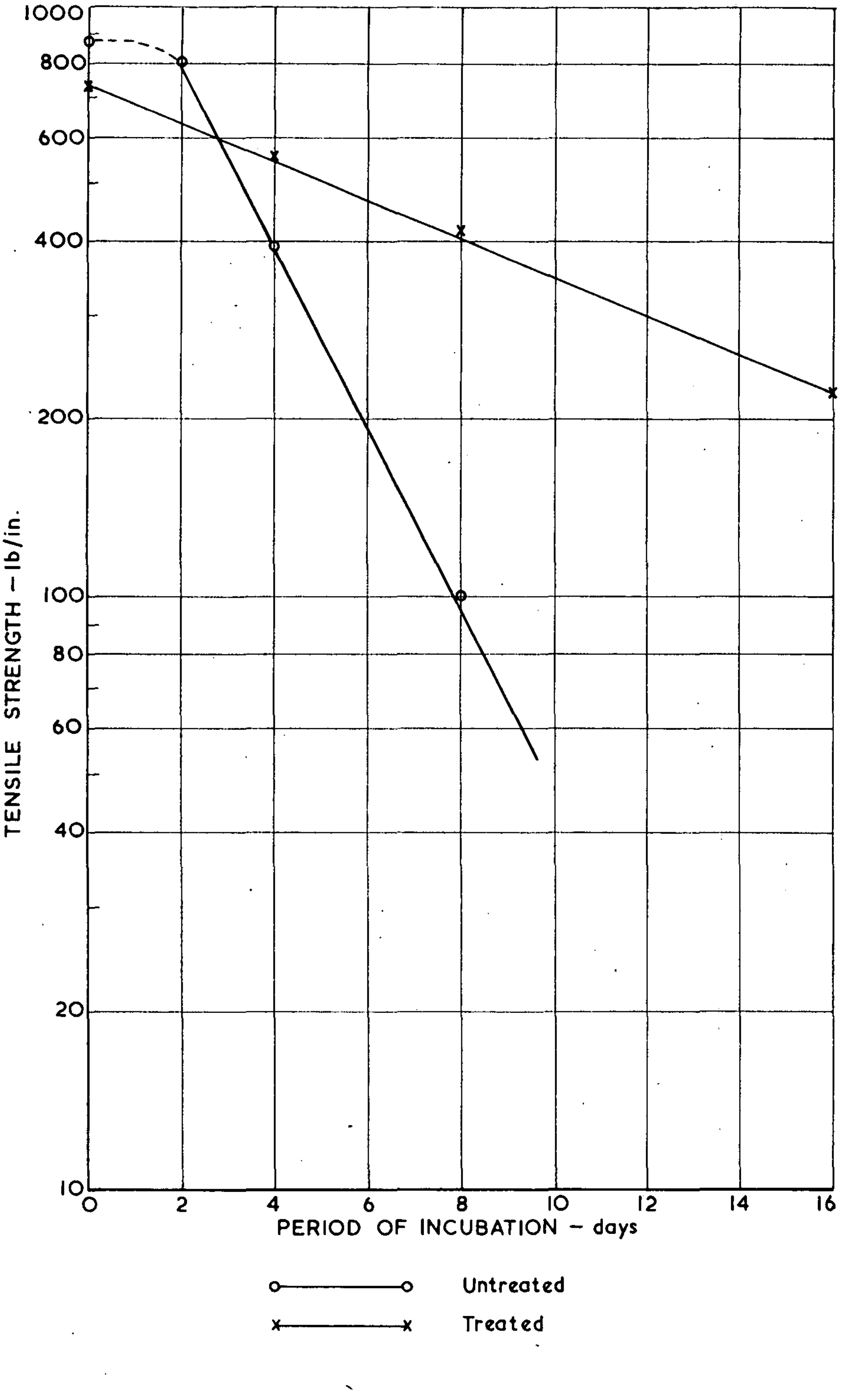

FIG. 2. THE VARIATION OF TENSILE STRENGTH WITH PERIOD OF INCUBATION. MAKE B.

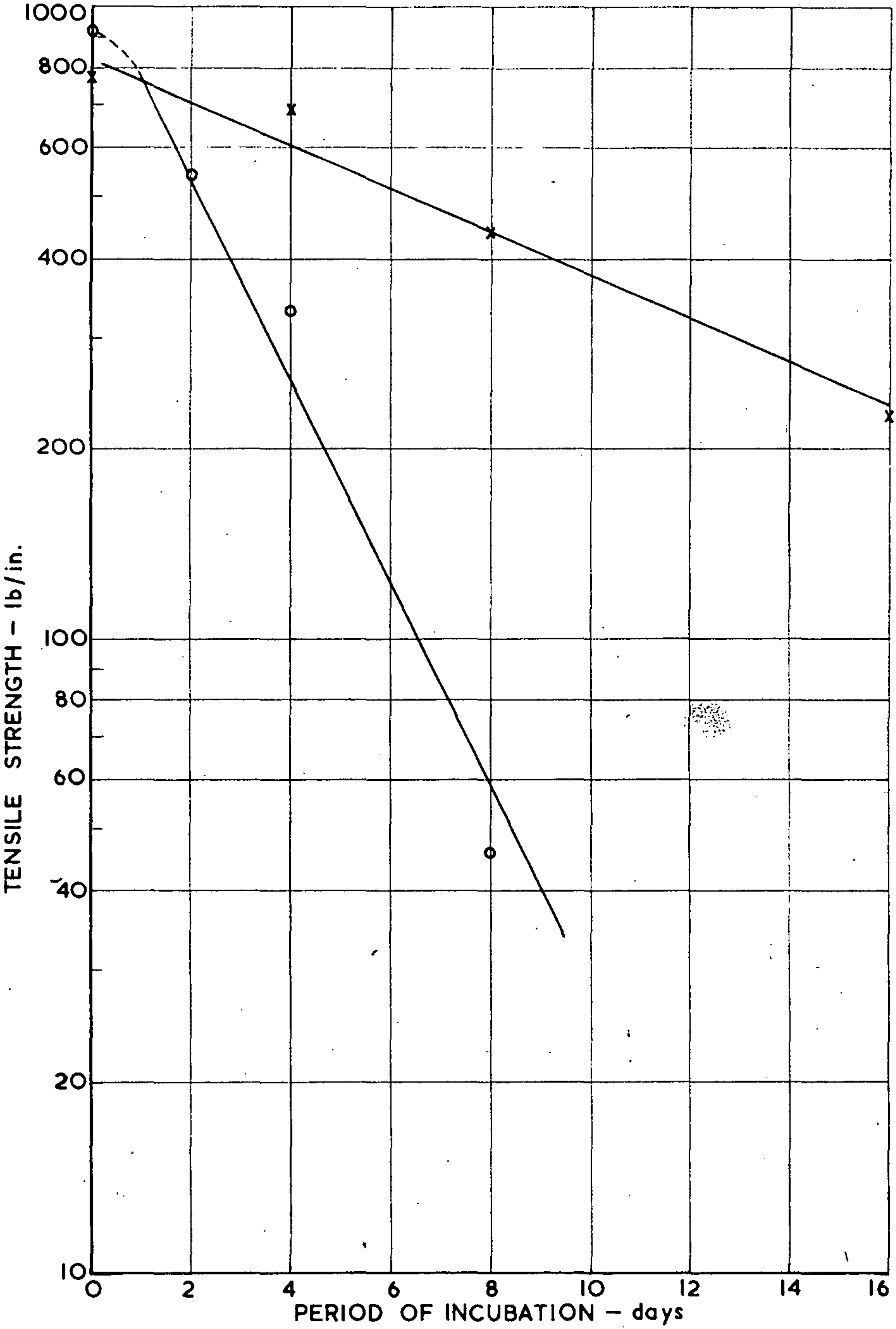

FIG. 3. THE VARIATION OF TENSILE STRENGTH WITH PERIOD OF INCUBATION. MAKE C.

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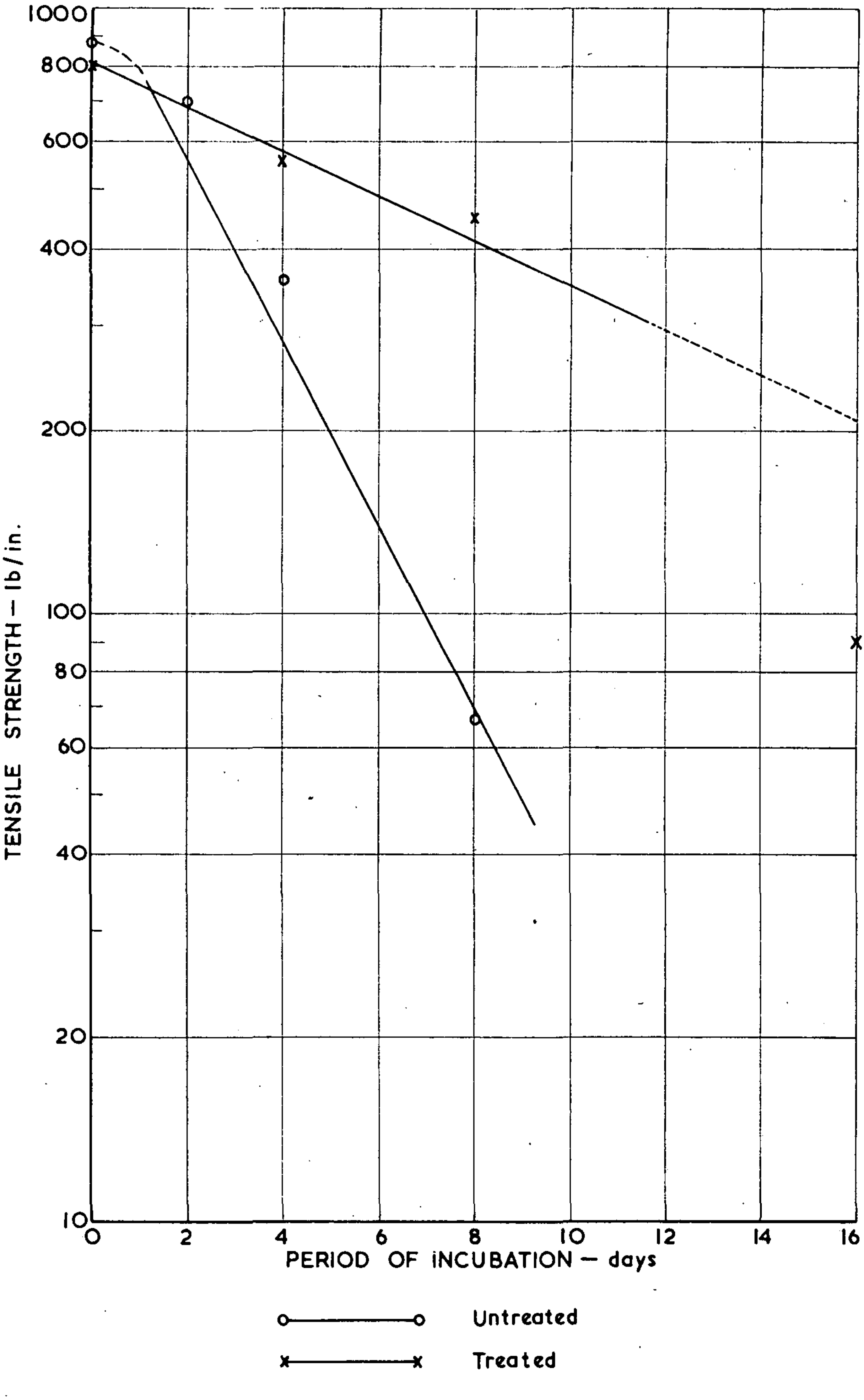

FIG. 4. THE VARIATION OF TENSILE STRENGTH WITH PERIOD OF INCUBATION. MAKE D.

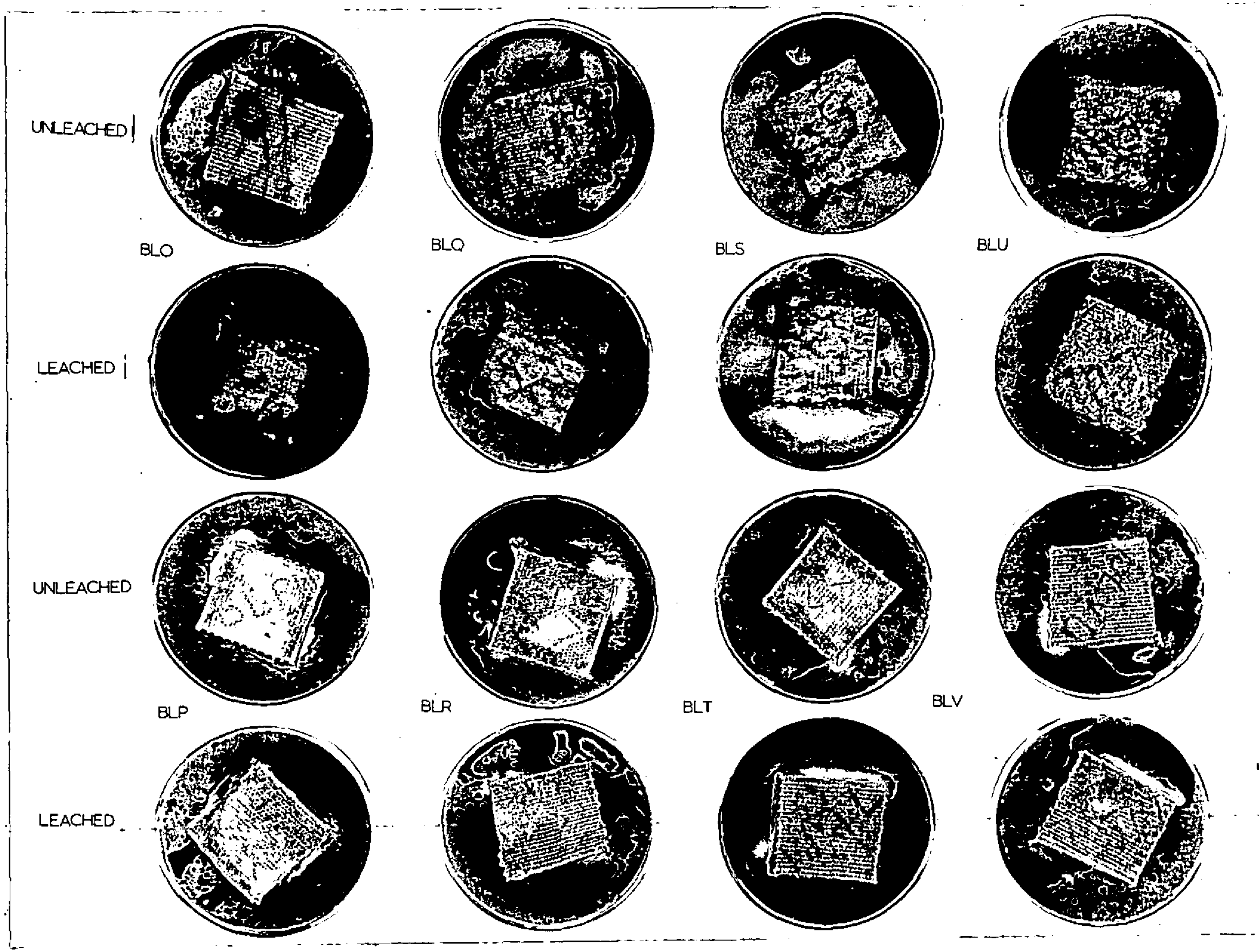

PLATE 1 - SPECIMENS OF HOSE AFTER INCUBATION WITH ASPERGILLUS NIGER.