

PRESERVATION OF WOOD.

PART I—TREATMENT WITH CREOSOTE-WATER EMULSION.

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Among the many preservatives used for protecting wood against natural decay and attack by fungi and insects, coal tar creosote is probably the best known and the most widely used. In the modern methods of treatment, the dried or seasoned timber is first subjected to a vacuum after preliminary treatment by steam, and creosote is then introduced. Under certain conditions—generally from 1.5–2 hours at 180°F. and under a pressure of 180 lbs./sq. inch—it is found that the requisite quantity of creosote is absorbed. The excess is then removed and the wood again subjected to a vacuum to draw off the surface oil.

It is found, in many cases, that the quantity of creosote absorbed is much more than is absolutely necessary for preservative purposes. Impregnation under a lower pressure or for a shorter time is no remedy, as it will result in incomplete penetration. It is, therefore, necessary to dilute the creosote if the quantity of it is to be reduced, maintaining the depth of penetration the same as with the pure material.

One of the diluents at present in use is what is commercially known as 'earth oil', a high-boiling petroleum fraction. Water is the obvious choice for a diluent from the point of view of cost and as it is not miscible with creosote, an emulsion of the two has to be used. The idea of using an emulsion is not new, quite a number of them having been described in patent literature; but comparatively large quantities of stabilizers have been used in most cases, with the possibility of interference with the action or the penetration of the preservative. More recently, a creosote emulsion called 'Plexosote' has been made by Messrs. Burt, Boulton and Haywood (China, *Proc. Chem. Eng. Group, Soc. Chem. Ind.*, 1924, 6A, 124) with the help of the Premier Colloid Mill and very good results are claimed for it.

Some species of wood absorb creosote only with difficulty even after prolonged treatment and it was expected that they would be more susceptible to penetration by an oil-in-water emulsion, as wood is wetted more readily by water than by oil. The present work was, therefore, undertaken with the two-fold object of reducing the cost of treatment and of securing better penetration in the case of refractory timbers.

EXPERIMENTAL.

Preparation of the Emulsion.—The emulsion of creosote-in-water suitable for wood-impregnation must be stable at 180°F. and under a

pressure of 180 lbs./sq. inch at which impregnation is carried out. Further, it must be absorbed by wood as such without being broken in its passage through the cell-walls. It is necessary to have as little of the stabiliser as possible.

The Premier Mill (China, *loc. cit.*) consists essentially of a conical-shaped rotor, moving in close proximity to a cup-shaped stator, the space between the rotor and stator being adjustable from about 0.01" to 0.001"; the rotor moves at about 15,000 revolutions per minute.

The emulsions used in the present study were prepared as follows:—Mixtures of creosote and aqueous solutions of the stabilizer were allowed to pass through the mill a number of times, when they were discharged as emulsions. The mixtures were fed by means of a funnel to the bottom of the machine and were forced through the narrow gap between the stator and rotor, the shearing action producing a very fine emulsion. The gap was kept at 0.003" in all the experiments. It was found in later experiments that a single passage through the mill was enough to give proper emulsification, 40 gallons being produced during one hour by a 5" mill. Practically all the emulsions prepared were of the oil-in-water type, containing 50 per cent. creosote. It may be mentioned here that it is important to add the creosote to the aqueous solution of the stabilizer and not *vice versa*, in order to get proper emulsification, especially when large scale manufacture is carried out.

Emulsions were prepared with various quantities (0.5–10%) of the following stabilizers:—(1) sodium hydroxide; (2) Mysore washing soap; (3) ammonium oleate; (4) gelatine; (5) gum arabic; (6) sodium silicate; (7) peptone; (8) dextrin; (9) Turkey Red Oil; (10) rosin soap; (11) casein in alkali; (12) Nekal A.E.M.*; and (13) triethanolamine. The earlier emulsification experiments were done with wood tar creosote from Bhadravati, while later, owing to the partial closure of the wood distillation plant at Bhadravati, coal tar creosote (B.E.S.A. Specification) was used instead. It was found that coal tar creosote was much easier to emulsify than wood tar creosote and required much smaller quantities of stabilizer.

Peptone, casein, triethanolamine, Nekal A.E.M. or a mixture of Nekal A.E.M. and casein gave fairly stable emulsions, when 0.5–2 per cent. of the stabilizer was used. Emulsions prepared with triethanolamine were not sufficiently stable towards electrolytes; triethanolamine and peptone were too costly for the manufacture of emulsions. From all points of view, Nekal A.E.M. was found to be the best of all the stabilizers investigated, both for wood tar and coal tar creosote emulsions. 50 per cent.

* Nekal A.E.M. is a mixture of 80 per cent. finely powdered glue and 20 per cent. Nekal BX—a sulphonated naphthalene derivative. It is a product of I. G. Farbenindustrie A.G.

emulsions of coal tar creosote and water prepared with 0.5 to 1 per cent. Nekal A.E.M. as stabilizer remained stable during several months and withstood prolonged boiling. The stability was improved by neutralizing the small quantity of free acid in creosote with 0.5 per cent. of sodium hydroxide. It may be mentioned in this connection that the stability of oil-in-water emulsions is increased if rendered slightly alkaline.

Small Scale (Laboratory) Impregnation Experiments.—Air was pumped into an autoclave containing small pieces of timber commonly in use in Mysore and filled with creosote or emulsion, until the required pressure was obtained. Heat was simultaneously applied and a temperature of 190°F. and a pressure of 4–11 atmospheres were maintained during a period of 0.5 to 3.5 hours. The weight of creosote or emulsion absorbed was determined from the increase in weight of the samples after impregnation.

The pieces used were all of the same size, 4" × 4" × 2" and in each case, comparative experiments were done both with coal tar creosote and with the emulsion. The results of the experiments are given in Table I. The last four species of timber, Jumble, Nandi, Rangi and Thadasal, are very hard and absorb creosote only with great difficulty.

TABLE I.

(a) *Balagi* (*Poeciloneuron indicum*, *Bedd.*)

(Very hard and valuable as timber for buildings, railway sleepers and telegraph or electric transmission poles.)

Time of Impregnation	Pressure in atmospheres	Wt. (in g.) before treatment	Wt. (in g.) after treatment	Oil or emulsion absorbed (in g.)	Absorption per c. ft. (lbs.)
<i>Creosote.</i>					
3.5 hours ..	8–9	585	606	21	2.5
3.5 " ..	11	510	595	85	10.1
1.5 " ..	11	577	606	29	3.5
1.0 " ..	11	595	635	40	4.8
<i>50 per cent. emulsion.</i>					
2.5 hours ..	10–11	532	656	124	14.8
2.0 " ..	6–7 for 1.5 hrs.	597	647	50	5.9
1.0 " ..	12–13 for 0.5 " "	575	650	75	8.9

(b) *Dhuma* (*Dipterocarpus indicus*, *Bedd.*)
(Soft wood ; contains resin.)

Time of Impregnation	Pressure in atmospheres	Wt. (in g.) before treatment	Wt. (in g.) after treatment	Oil or emulsion absorbed (in g.)	Absorption per c. ft. (lbs.)
<i>Creosote.</i>					
3.5 hours ..	11	424	573	149	18.0
3.0 " ..	10	395	530	135	16.1
1.5 " ..	11	418	545	127	15.1
1.0 " ..	7	426	504	78	9.3
0.5 " ..	11	424	481	57	6.8
0.5 " ..	7	392	454	62	7.4
0.5 " ..	4	429	479	50	5.9
<i>50 per cent. emulsion.</i>					
2.5 hours ..	10-11	405	591	186	22.1
2.0 " ..	6-7 for 1.5 hrs. 12-13 for 0.5 "	398	610	212	25.2
1.0 " ..	11	404	562	158	18.8
0.5 " ..	11	450	584	134	15.9
0.5 " ..	4	446	530	84	10.0

(c) *Hunal* (*Terminalia paniculata*, *W. and A.*)

(Very hard and fairly durable ; used for posts, beams, rafters and planking.)

<i>Creosote.</i>					
3.5 hours ..	11	399	521	122	14.5
3.5 " ..	8-9	483	560	77	9.2
1.5 " ..	11	451	540	89	10.6
1.0 " ..	7	368	431	63	7.5
0.5 " ..	11	402	471	69	8.2
0.5 " ..	7	413	475	62	7.4
0.5 " ..	4	418	469	51	6.1

(c) *Hunal* (*Terminalia paniculata*, *W. and A.*)—*Contd.*

50 per cent. emulsion.					
2.5 hours ..	10-11	420	562	142	16.9
2.0 " ..	6-7 for 1.5 hrs. 12-13 for 0.5 "	442	577	135	16.1
1.0 " ..	11	424	527	103	12.3
0.5 " ..	11	454	529	75	8.9
0.5 " ..	4	438	497	59	7.0

(d) *Kanagal* (*Dillenia pentagyna*, *Roxb.*)
(Moderately hard, heavy, strong and durable.)

<i>Creosote.</i>					
3.5 hours ..	11	396	465	69	8.2
3.0 " ..	10	479	521	42	5.0
1.5 " ..	11	408	444	36	4.3
1.0 " ..	11	374	417	43	5.1
50 per cent. emulsion.					
3.0 hours ..	11	420	508	88	10.5
1.0 " ..	11	400	472	72	8.6

(e) *Malhi* (*Terminalia tomentosa*, *W. and A.*)
(Very hard and close-grained; used for structural timber.)

<i>Creosote.</i>					
3.5 hours ..	11	516	593	77	9.2
3.0 " ..	10	413	534	121	14.4
1.5 " ..	11	523	574	51	6.1
1.0 " ..	11	426	489	63	7.5
50 per cent. emulsion.					
3.0 hours ..	11	541	655	114	13.6
1.0 " ..	11	500	563	63	7.5

(f) *Yethiaga* (*Adina cordifolia*, *Hook f.*)
 (Lemon-yellow and moderately hard; used for making furniture,
 combs and toys.)

Time of Impregnation	Pressure in atmospheres	Wt. (in g.) before treatment	Wt. (in g.) after treatment	Oil or emulsion absorbed (in g.)	Absorption per c. ft. (lbs.)
<i>Creosote.</i>					
3.5 hours ..	11	386	550	164	19.5
3.5 " ..	8-9	360	506	146	17.4
1.5 " ..	11	425	574	149	17.7
1.0 " ..	7	406	524	118	14.0
0.5 " ..	11	418	535	117	13.9
0.5 " ..	7	407	521	114	13.6
0.5 " ..	4	396	497	101	12.0
<i>50 per cent. emulsion.</i>					
3.0 hours ..	10-11	388	581	193	23.0
1.25 " ..	7-8	372	536	164	19.5
1.0 " ..	11	420	520	100	11.9
0.5 " ..	11	414	588	174	20.7
0.5 " ..	4	407	510	103	12.3

(g) *Jumbe* (*Xylia dolabriformis*, *Benth.*)
 (Tough, strong and very hard; useful for making posts, beams
 and scantlings.)

<i>Creosote.</i>					
0.5 hour ..	5	213	232	19	4.5
<i>50 per cent. emulsion.</i>					
0.5 hour ..	5	208	234	26	6.2
0.5 " ..	5	224	244	20	4.8

(k) *Nandi* (*Lagerstroemia lanceolata*, *Wall.*)
 (Moderately hard, elastic and tough; used for building purposes.)

Time of Impregnation	Pressure in atmospheres	Wt. (in g.) before treatment	Wt. (in g.) after treatment	Oil or emulsion absorbed (in g.)	Absorption per c. ft. (lbs.)
<i>Creosote.</i>					
0.5 hour ..	5	162	167	5	1.2
0.5 " ..	5	166	172	6	1.4
<i>50 per cent. emulsion.</i>					
0.5 hour ..	5	159	170	11	2.6
0.5 " ..	5	167	179	12	2.9

(l) *Rangi* (*Mimusops elengi*, *Roxb.*)
 (Heavy, strong and durable; used for house-building.)

<i>Creosote.</i>					
0.5 hour ..	5	228	240	12	2.9
0.5 " ..	5	230	244	14	3.3
<i>50 per cent. emulsion.</i>					
0.5 hour ..	5	223	242	19	4.5
0.5 " ..	5	233	249	16	3.8

(m) *Thadasal* (*Grewia tilliaefolia*, *Vahl.*)
 (Very elastic and moderately hard; used for cart and carriage building.)

<i>Creosote.</i>					
0.5 hour ..	5	173	210	37	8.8
<i>50 per cent. emulsion.</i>					
0.5 hour ..	5	172	258	86	20.5
0.5 " ..	5	174	238	64	15.2

It is seen from the results that, in most cases, absorption of emulsion is much larger than, and sometimes twice as much as, creosote itself, temperature, pressure and period of impregnation being the same. The interpretation of the results is rather difficult, as there is a very large variation in the absorptive capacity not only of the different species treated, but also of parallel samples of the same species treated under exactly similar conditions. The former is, of course, well-known and perfectly understandable; the latter is also to be expected as wood, being a non-homogeneous material, varies from sample to sample containing different proportions of heartwood, although not so largely as between one species from another.

One generalization can, however, be made from the results: that the emulsion is absorbed much more easily than creosote. Mathi is probably an exception. Interior sections of the samples were examined to measure the depth of penetration by the antiseptic; but it was found that in all the cases, the penetration was complete, so that it was not possible to compare the rates of penetration of creosote and emulsion. Larger pieces of wood will be used in future experiments and the variation of pressure and period of impregnation will be arranged to be much less, so that more comparable data can be obtained.

The foregoing experiments conclusively proved the stability of the emulsion on standing and to boiling; and more important still, they have shown that the emulsion is not broken down by the cell-walls of the wood in its passage through them. In fact, the same emulsion was used for a number of experiments without undergoing any appreciable separation.

Experiments on a semi-commercial scale.—This part of the work was carried out on a fairly large scale at the Creosoting Plant, Bhadravati. An experimental impregnating cylinder of about 500 gallons capacity, specially constructed for the purpose, was used.

50 per cent. emulsions were used as before, but the quantity of stabilizer was reduced to 0.5 per cent. Large pieces of wood such as scantlings and metre gauge railway sleepers were used and each experiment required about 300 gallons of emulsion. The results are summarised in Table II.

It will be seen that the results of the small scale experiments are, in the main, confirmed by those carried out on a semi-commercial scale, allowing for exceptions. Owing, however, to the fact that wood is far from being a homogeneous material, a much larger number of experiments will have to be done with each species of wood and the average taken, if quantitative data are to be obtained. Moreover, as heartwood absorbs very little creosote, the total absorption of a sample is dependent chiefly on the proportion of sapwood it contains. This explains the discordant results. At best, one can get only a rough idea of the order of absorption of the emulsion.

TABLE II.

Species of wood treated	Wt. before treatment (in lbs.)	Wt. after treatment (in lbs.)	Absorption of emulsion (in lbs.)	Cubical contents (in c. ft.)	Absorption per c. ft. (in lbs.)	Absorption of creosote per c. ft. at 90 lbs./sq. inch and 180° F.	Absorption of creosote per c. ft. at 180 lbs./sq. inch and 180° F.	Remarks
1. Dhuma, M. G. Sleeper ..	103.25	111	7.75	1.5	5.2	3.8	6.5	180° F.
2. " " ..	85.5	92	6.5	1.5	4.3	3.8	6.5	Initial vacuum 25" for 20 minutes.
3. " " ..	81.5	88.75	7.25	1.5	4.83	3.8	6.5	
4. Mathi, T. G. Sleeper ..	71.25	76.25	5.0	1.0	5.0	3.9	5.0	90 lbs./sq. inch for one hr.
5. " " ..	63.5	67.5	4.0	1.0	4.0	3.9	5.0	Initial vacuum 25" for 15 minutes.
6. " M. G. Sleeper ..	100.8	103.5	2.7	1.5	1.8	3.9	5.0	
7. " 7' x 3" x 2" ..	292.5	309.3	16.8	4.4	3.8	3.9	5.0	180 lbs./sq. inch for 1 hr.
8. " Sizes ..	1126.3	1204.5	78.2	16.4	4.8	3.9	5.0	Temperature 180° F. Initial vacuum 25" for 30 minutes. Pressure 100 lbs./sq. inch for 1.5 hrs.
9. " Sleepers ..	227.3	262.5	35.2	4.0	8.8	3.9	5.0	Do. and final vacuum 25" for 0.5 hour.
10. Hunal, T. G. Sleeper ..	60.8	65.8	5.0	1.0	5.0	3.0	4.5	
11. " " ..	63.3	67.8	4.5	1.0	4.5	3.0	4.5	

TABLE II.—(Contd.)

Species of wood treated	Wt. before treatment (in lbs.)	Wt. after treatment (in lbs.)	Absorption of emulsion (in lbs.)	Cubical contents (in c. ft.)	Absorption per c. ft. (in lbs.)	Absorption of creosote per c. ft. at 90 lbs./sq. inch and 180° F.	Absorption of creosote per c. ft. at 90 lbs./sq. inch and 180° F.	Remarks
12. Huna, M. G. Sleeper ..	101.3	103.8	2.5	1.5	1.7	3.0	4.5	Temperature 180° F. Initial vacuum 25" for 0.5 hour.
13. „ 7'×3"×2" ..	257.8	310.0	52.2	4.4	11.9	3.0	4.5	
14. Balagi, Sizes 2'×2.3"×5" ..	59.3	66.8	7.5	0.5	14.1	5.5	7.5	
15. „ ..	53.3	63.5	10.2	0.5	19.2	5.5	7.5	
16. Nandi, 8¼'×6"×3" ..	294.8	364.8	70.0	6.2	11.3	..	4.5	
17. „ 7'×3"×2" ..	176.5	197.3	20.8	3.5	5.9	..	4.5	
18. Yethiaga, M. G. Sleeper	101.3	104.0	2.7	1.5	1.8	..	12.5	
19. Kanagal, „ ..	96.3	104.3	8.0	1.5	5.3	..	6.5	
20. „ „ ..	77.8	110.0	32.2	1.5	21.5	..	6.5	

In order to find out the comparative rates of penetration, experiments will be done with larger pieces at lower pressures so that there is incomplete penetration of the antiseptic. Another very important point to be decided is whether the emulsion is absorbed uniformly as such or there is preferential absorption of water or creosote from the emulsion. Such selective absorption is possible although, as has been found out, the emulsion is not broken in its passage through the cell-walls of wood. In order to settle this point, it is necessary to devise a method for estimating creosote in the treated wood. Experiments in this direction are on hand.

Finally it may be mentioned that the emulsion-treated samples of wood have been allowed to lie exposed during two years and have shown no signs either of insect or fungus attack. But, it should be noted that, in all such experiments, the only satisfactory test is that of time.

SUMMARY.

A stable 50 per cent. emulsion of coal tar creosote in water suitable for wood-impregnation has been successfully prepared, using 0.5-1 per cent. Nekal A.E.M. as stabilizer.

Laboratory and large scale impregnation experiments conducted with this emulsion on a number of well-known Indian timbers have shown that the emulsion is absorbed without being broken down by the cell-walls of the wood and a much larger quantity of the emulsion than of creosote is absorbed, under the same conditions of temperature and pressure.

Emulsion-treated samples of wood exposed during the last two years have resisted insect and fungus attack.

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