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MODIFICATIONS OF SHELLAC

Part I

THE EFFECT OF SULPHUR

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THE AIM OF THE INVESTIGATION, the results of which are described in the following pages, has been to improve the properties of shellac so that it may find greater application in the industries where it is largely consumed at present. The industries, with which it is chiefly concerned, are electric, moulding and varnish industries. In all these shellac has been able to maintain its position mainly by virtue of its certain peculiar properties such as good insulating qualities, thermoplasticity and capacity for taking a good polish combined with other desirable properties like hardness, toughness, resistance to abrasion, etc. In spite of these properties, shellac has two important disadvantages, namely, (1) its low softening point and (2) its non-resistance to water, which act as limitations for its further application. The object of the present investigation is, therefore, not only to improve these defects of shellac but also to alter its properties in general by suitable modifications such as is generally done in synthetic resins so as to meet the needs of the various consumers. One suitable modifying medium has been found in sulphur, and its organic and inorganic compounds. A few preliminary experiments showed that sulphur in its pure elemental form when fused with shellac within certain limits has been able to impart improved properties with regard to its degree of hardness and resistance to water. Work was therefore undertaken to study in more detail the optimum conditions with regard to time and temperature of fusion, percentage of sulphur that could be effectively combined, etc., and to examine the properties of the resultant products.

Though nothing is mentioned in the literature of the effect of sulphur and its compounds on lac and shellac, there are ample references to be found in connection with their use for improving the properties of some natural and synthetic resins and drying oils. The use of sulphur in the vulcanisation process of rubber is sufficiently well known. One of the earliest investigators on the action of this element on natural resins was Zingler (29) who prepared sulphuretted copal by heating 50 kg. of powdered gum copal with $7\frac{1}{2}$ -15 kg. of flowers of sulphur. About nine years later another chemist Valenta (25) obtained by adding sulphur to molten rosin a clear mass of sulphuretted product which on further heating turned brown and gave off hydrogen sulphide. In 1915, Prager (15), by combining 5-10% of sulphur with rosin at 140°-150°C, produced a dark green product quite resistant to the action of weather or chemical reagents. Two years later he was followed by Low (12) who took out a patent for obtaining hardened products of improved resistance to oxidation from the natural resins like rosin, manila copal, dammar, etc., by heating them under pressure with an aqueous suspension of sulphur. Between the years 1928-29 Auer (2)

took a series of patents for modifying the physical properties of rosin and ester gum with the use of sulphur and its metal-free organic and inorganic compounds, under various conditions of treatment for the production of water-fast varnishes and lacquers. Quite recently two more patents have been taken by Dörken (6) and Scheiber (21) for modifying the properties of resins and varnishes in general with the aid of sulphur and its compounds.

Much work has also been done in altering the properties of synthetic resins with this reagent. As early as 1887 Tassinari (23) observed that a resinous product could be obtained by reacting phenol with sulphur chloride. Recent investigations on these resins were carried out by Ellis (7) who found that the properties of such resins varied with the proportion of sulphur chloride used. The larger the proportion of sulphur chloride to phenol, the harder, more infusible and less soluble were the resultant resins. Mc. Coy (13) used Tassinari's resin in conjunction with phenolformaldehyde products. The well-known processes of "factis" manufacture and the vulcanisation of rubber are also condensations with sulphur chloride, in which the raw materials are mainly natural products such as fatty oils and india rubber. Corresponding treatments are also given to other artificial resins like anhydroformaldehyde-aniline, coumarone resins, aldehyde resins, polymerides of vinyl resins, and so forth (22). Arsem (1) obtained a vulcanised product of glycerylphthalate resin which had qualities somewhat resembling rubber. Several condensation products containing sulphur have been made more recently by Carpmael (4) by converting oils to resin-like condensation products containing the residues of polyvalent alcohols, polybasic acids and unsaturated carboxylic acids by reaction with sulphur or agents providing sulphur. Solutions of these in volatile solvents are stated to yield, after drying, lustrous coatings which are stable to sodium carbonate. A substance used for impregnating and known under the trade-name of "Toron" is produced in America from turpentine and sulphur (16), (8).

Greater attention has also been paid to the vulcanisation of drying and semidrying oils and their varnishes with a view to improving their properties. Barringer (3) finds that addition of sulphur to the product obtained by heating the oils with zinc oxide or hydrated lime at 200°C makes it unattackable by water. Anderson, Altschul and Henriques studied the properties of sulphurised linseed oil as far back as 1895, but it is only during later years that it has come into use industrially. Several publications of late have appeared on the investigation of these oils, of which the studies of Höllander (11), and Salvaterre and Suda (20) merit special attention. These authors claim that by combining sulphur with linseed oil under suitable conditions, the dried films from such oil may be made more water-resistant, comparable to those obtained from polymerised tung or stand oils. The work of these investigators has been extended to the study of other oils such as rape seed oil, soyabean oil, poppy seed oil, castor oil, etc., by Gge (10) who recommends that such sulphurised oils are especially suitable for the protection of automobile tyres against moisture. With regard to the protective action of sulphur on varnishes, Gardner and Sward (9) in America have made some exposure tests on the durability of certain synthetic resin oil varnishes containing the optimum content of sulphur, and find such varnishes stand weathering tests better than others.

From the above brief review of the work done, the importance of sulphur or its compounds in beneficially modifying or altering the properties, more particularly those of water resistance and hardness, of the various natural and synthetic resins and varnishes will be quite apparent. Since none of the investigators so far reviewed have referred to any work of this nature on lac or shellac, it was thought desirable to study the effect of sulphur on this resin in all its aspects.

Two short notes (26), (27) describing the effects of sulphur treatment have already been published. In this paper a more detailed description of the results obtained will be given.

EXPERIMENTAL

Several methods are recommended for sulphurising resins and similar substances: (1) by direct fusion with sulphur in the dry condition with and without the aid of catalysts; (2) by heating under pressure with an aqueous suspension of sulphur; (3) by the aid of inorganic compounds of sulphur, like sulphur chlorides, carbondisulphide, etc. Methods (1) and (2) were first investigated and efforts made to study the optimum conditions of reaction. The resulting products were examined for such specific properties as acid and iodine values, and their varnishing qualities. Some preliminary experiments conducted with sulphur chlorides as mentioned in the last method showed that the reaction was too rapid to control and the lac became polymerised long before any combination took place. The reaction with this reagent, however, can be controlled by carrying out the experiments in the cold in the presence of solvents. A note (27) on this last aspect of the investigation has been published.

Experiment I. By direct fusion in air.— Pure *Schleichera trijuga* (Kusum) shellac and flowers of sulphur were selected for the purpose of this investigation. During experiments with the fusion of shellac with sulphur it is noticed that the properties of the resulting products are greatly dependent upon the quantity of sulphur used, the duration of heat employed and the temperature at which the reaction is conducted. For instance, it is observed that if the percentage of sulphur is increased beyond a certain minimum or if the temperature and duration of reaction are unduly prolonged, the resulting product becomes more brittle and excess of uncombined sulphur remains over. Optimum conditions of working were, therefore, first investigated. Three different temperatures, *viz.*, 150°C, 170°-180°C and 200°C were selected and at each of these, mixed samples of shellac and sulphur were heated over an oil bath for different periods in order to find the optimum time of fusion and rate of reaction without the risk of samples getting "cured" or polymerised. It was observed that at 150°C, the reaction was very slow, the lac getting polymerised long before proper combination had taken place, and at 200°C, the reaction was very rapid, besides the resulting product on cooling became more brittle. Fusion conducted at the mean of these two temperatures, *viz.*, 175°-180°C, was found to be suitable in that the experiment could be completed within 30-40 minutes, and the final products also when cooled had the proper degree of hardness and colour. For the subsequent experiments, therefore, the latter conditions of temperature and time were used. It will be seen that the temperature used closely agrees with the one employed by Carpmael (*loc. cit.*) in his experiments with sulphur on condensation products of polyvalent alcohols, polybasic acids and unsaturated carboxylic acids.

About 25 grams of powdered shellac, mixed with varying percentages of sulphur, were heated over an oil bath at the stipulated conditions of time and temperature with frequent stirring and the melt was allowed to cool on galvanised iron sheets. During heating it was observed that the reaction went on slowly in the beginning and after some time it became vigorous with rapid bubbling and evolution of gas and moisture. The gas which evolved was identified to be hydrogen sulphide which showed that the sulphur might have entered into some sort of chemical combination. The ultimate products also were dark green in colour and possessed a slight sulphur-like odour (*cf. Prager, loc. cit.*) The final products were then examined for their properties.

The amount of hydrogen sulphide that is actually formed during the reaction under the conditions of the experiment was estimated in a few samples according to the method described by Treadwell and Hall (24). The results obtained indicated that the amount of sulphur removed as hydrogen sulphide was small, being of the order 1-2% of the weight of sulphur present.

Experiment II. By fusion under pressure.—A few samples of powdered shellac were heated with an aqueous suspension of sulphur in an autoclave under a pressure of 3-4 atmospheres, as is generally employed in the vulcanising process of rubber. The temperature attained at this pressure was about 150°C, which, though low judging from the conditions needed for dry fusion, was found to be sufficient for the proper combination of sulphur. The ultimate product also was dark green in colour as was observed in the case of direct fusion, but it was found that the properties were not so good as those of the products obtained by the first method. Nevertheless, the process possessed an advantage in that the samples could be heated for any length of time without the fear of polymerising them. The products obtained by this method were also examined for their properties and compared with those resulting from the first method.

RESULTS & DISCUSSION

I. Properties : (a) Effect of water.—4 lb. cut varnishes of all the samples in specially denatured alcohol were prepared in the cold and filtered to remove wax and insolubles. A difficulty was, however, experienced in filtering such thick viscous solutions rendered more colloidal by heat, but this could be overcome by mixing the solutions with kieselguhr or any similar filter aids, prior to filtration. The filtered varnishes were all greenish yellow in colour. Films of uniform thickness from these varnishes were prepared on glass slides by the spinning wheel method. All the films were dried for ten days at the ordinary room temperature before examining them. On one set of films, the resistance to water and water-absorbing capacity were measured. The results of these tests are presented in the following table.

TABLE I
WATER ABSORPTION AT 26°C

SAMPLE	24 HRS.	APPEARANCE	48 HRS.	APPEARANCE
	per cent		per cent	
Control, original unheated ..	14.7	Partially transparent	20.1	Opaque
Control, heated ..	11.2	" "	16.3	Partially opaque
Shellac + 1% sulphur	10.4	" "	14.5	" "
" + 2% " ..	5.4	Transparent ..	7.7	Transparent
" + 3% " ..	7.4	Partially transparent	11.1	Partially transparent
" + 4% " ..	4.9	Transparent ..	6.4	Transparent
" + 5% " ..	6.2	" ..	7.4	"
" + 10% " ..	7.8	Milk white opalescent	10.8	Opalescent
Control, autoclaved ..	19.8	Completely opaque ..	25.1	Completely opaque
Shellac + 4% sulphur autoclaved ..	7.8	Partially transparent	12.4	Partially transparent

From the figures given in the table, it will be seen that the optimum percentage of sulphur which could be incorporated in lac lies in the range between 4-5%, the water absorption thereby being reduced to one-third the value given by control. Higher percentage of sulphur, for example, 10% sulphur, increases slightly the water absorption as is evident from the figures shown in the table. Sulphurising lac in the wet way under pressure in an autoclave does not show any special advantage over the direct fusion method, but even so it will be seen that the sample containing 4% sulphur gives only half the figure for water absorption than the one given by the sample treated under the same conditions, but without sulphur. All the films including the control gave off completely the water absorbed within 24 hours, thereby showing the water absorption as only a physical phenomenon. A remarkable improvement in the resistance of the films to water could be got by baking the films for a short period at 90°C.

(b) **Scratch hardness and resistance to abrasion.** — On a second set of films prepared on glass slides, scratch hardness and resistance to abrasion were then tested before and after baking. The details of carrying out these tests have already been described in a previous bulletin of this Institute (17). Table II shows the results obtained, the mean of several readings having been given.

TABLE II
SCRATCH HARDNESS & ABRASION FACTOR

Sample	SCRATCH HARDNESS		ABRASION FACTOR	
	Before baking	After baking at 100-105°C for 3 hours	Before baking	After baking at 100-105°C for 3 hours
Control original unheated ..	400 g.	2725 g.	246	346
Control, heated	490	2115	270	1286
Shellac + 1% sulphur	360	2525	250	2755
" + 2% "	400	2700	287	2050
" + 3% "	420	3725	237	2835
" + 4% "	560	2420	400	6010
" + 5% "	560	2825	390	4540

The advantage of incorporating sulphur with shellac with regard to its scratch hardness and resistance to abrasion will be obvious. The optimum percentage of sulphur that could be beneficially combined lies again between 4-5%. It will also be seen that slight baking of the films improves enormously the wearing qualities of the sulphur-treated resin films. The improvement in properties as the result of baking, on shellac films in general has already been reported by Rangaswami and Aldis (18).

(c) **Elasticity and adhesion.** — These two factors were studied qualitatively with films coated on strips of tin sheets by the usual bending test methods. Each specimen after drying for about ten days is bent double rapidly over a $\frac{1}{2}$ " iron rod at the temperature of the laboratory

and then the bent portions of the films are examined for any cracks or peeling. The results of these tests are summarised in the following table, which represents the mean of several tests.

TABLE III
BENDING TESTS ON THE SULPHUR-TREATED RESIN FILMS

SAMPLE	OBSERVATION
Control ; original unheated	Long parallel cracks, poor adhesion.
Control ; heated	Very slight cracking, fair adhesion.
Shellac + 1% sulphur	No cracking, good adhesion.
.. + 2%	" "
.. + 3%	" "
.. + 4%	Slight cracking, poor adhesion.
.. + 5%	Long close parallel cracks ..
.. + 10%	" "

N.B. — All the tests described under (a), (b) and (c) were completed in one day to prevent the influence of change in the conditions of humidity and temperature.

From the observations made on these tests it will be clear that samples treated with 1-3% sulphur show an improvement with regard to their elasticity and adhesion in that the films did not crack or peel off from the test piece.

II. (a) Action of colour on sulphuretted shellac and its varnishes. — It has already been said that the final reaction products of shellac and sulphur and also the varnishes prepared from them are greenish in colour. Though the colour of these products is green it has been found that they take up any pigment or spirit-soluble dye remarkably well giving bright coloured objects. Ordinarily, to colour shellac or its varnish they will have to undergo the costly process of bleaching beforehand or require fairly large quantities of pigments or dye stuff. It has been found that sulphuretted shellacs require only small quantities of colouring principles, and a definite estimation showed the actual requirements to be less than a quarter the amount usually needed for straight shellac samples. Besides, the sulphur in shellac increases the covering power of the dye stuffs. It may also be mentioned that the coloured objects prepared from ordinary shellac are not so good in that they all lack in brightness, shade and tint. In view of the ease with which these sulphur-treated samples take up any colour, and their low cost of manufacture together with the possession of certain qualities peculiar to these bodies such as resistance to water, hardness, ability to withstand wear, etc., on account of their content of sulphur, adaptation in due course to the various industrial uses may be anticipated. Several bright coloured varnishes have been prepared in the laboratory and objects like wood and glass have been coated and examined. Among the numerous applications where they can be profitably applied, a few examples like the manufacture of coloured toys, coating electric bulbs, lamp shades, window panes, etc., may be mentioned.

(b) **Preparation of coloured mouldings.** — The possibility of preparing coloured mouldings from these sulphurised shellacs has also been tested, and several moulds in green, blue, pink, red and mottled shades have been prepared and examined. All the samples have retained their bright shades even after nearly twelve months of their preparation. The properties thus exhibited by these show an improvement over the ordinary shellac. Further, the mouldings obtained from the sulphurised samples have been found to possess greater water resistance and impact strength than those prepared from untreated shellac. In view of the above properties it is suggested that several industrial applications may be made for the production of fancy coloured mouldings of various designs such as brush handles, clock cases, coloured gramophone discs, bottle caps, etc., all of which, in general, do not require any great heat resistance.

(c) **Resistance to surface carbonisation.** — Another important property which is concerned chiefly with the electrical insulating qualities of these moulded samples, namely, the resistance to surface carbonisation by sparking or arcing has also been studied. The surface carbonisation, or what is technically known as 'tracking' has been the serious electrical defect of bakelite mouldings (14), (28), (5). It is observed that the sulphur-treated shellac mouldings have been able to withstand this tendency to 'tracking' much better than even shellac mouldings, presumably, due to the presence of the strong insulating material, sulphur.

III. Chemical properties. — In addition to the industrial properties described in the foregoing a few physical and chemical properties of the sulphurised shellac samples have also been examined with a view to finding any change in the constitution or chemical composition which might have been undergone as the result of the treatment. Evolution of hydrogen sulphide during the reaction and the gradual change in colour of the resin undoubtedly indicated some kind of chemical reaction with sulphur entering into the constitution. Since the alcoholic solution of the resin is also green in colour, it is possible that sulphur might have reacted with erythrolaccin, the colouring principle of lac.

As the first step towards the examination of these samples it was thought desirable to estimate the amount of insolubles in hot alcohol in order to find if any unreacted sulphur is left over. The procedure adopted for these estimations is the same as the one employed by the United States Shellac Importers Association for insolubility determinations in shellac. The results are tabulated below :

TABLE IV

INSOLUBLES IN SULPHUR-TREATED SHELLAC

Control heated	0.65 per cent
Shellac + 1% sulphur	0.88 " "
.. + 2%	0.80 " "
.. + 3%	0.80 " "
.. + 4%	0.87 " "
.. + 5%	0.91 " "

From the figures it will be seen that the amount of insolubles remains fairly constant irrespective of the amount of sulphur used. This may be explained in two ways : (1) the sulphur might have formed a soluble compound with shellac, or (2) it might have changed into a soluble form

due to the heat treatment. To settle this point estimations of insolubles in the cold were made on two samples, one treated with the maximum percentage of sulphur, and the other without any sulphur. The results obtained are :

TABLE V

			Cold insolubles
Control shellac heated without sulphur	2.04 per cent
Shellac + 5% sulphur	2.82 " "

the increase of 0.78% cold insolubles in the case of the sulphur-treated sample being probably due to the amount of uncombined sulphur. From these figures it will be clear that at least 4% of sulphur must have reacted with shellac in the sample examined.

It may be mentioned here that the amount of hydrogen sulphide evolved is so small that it cannot account for any appreciable removal of sulphur.

Two other principal properties, namely, acid and iodine values, where any chemical change can be easily detected have also been studied. The results are given below. These results are obtained from samples from which any uncombined sulphur has been removed by cold filtration from their alcoholic solutions.

TABLE VI

ACID & IODINE VALUES OF SULPHUR-TREATED SHELLAC

	A. V.	I. V. (Wijs).
Control : original shellac	79.4	14.9 per cent
" " " heated without sulphur	78.1	16.4 " "
" " with 1% sulphur	78.9	20.8 " "
" " 2% " "	77.9	24.3 " "
" " 3% " "	78.2	28.8 " "
" " 4% " "	79.1	29.3 " "
" " 5% " "	78.8	34.0 " "
" " 10% " "	79.0	40.7 " "
Original shellac autoclaved at 3 atoms. for 3 hours	80.6	19.0 " "
" " " + 4% sulphur	82.1	31.4 " "

From the figures given it will be evident that practically no change has been brought about in the acid values, but there is a rapid increase in the iodine values. Addition of 4-5% sulphur actually doubles the iodine value the cause of which is however difficult to interpret. It is possible that during the melting operation substitution and splitting take place, introducing new double bonds in the shellac resin. The evolution of hydrogen sulphide is evidence for this possibility.

It is possible, however, that a reaction takes place between the loosely combined sulphur itself and the iodine. Efforts to determine the iodine value of flowers of sulphur gave a value of 18-20% which is, of course, insufficient to explain the effect, but it was obvious that a considerable proportion of the sulphur was unattacked under the conditions of the experiment.

In the sulphur-treated shellacs the sulphur is probably in a more available condition and it would not be unreasonable to assume that it can have a higher iodine value in these circumstances. A similar observation regarding the increase of iodine value brought about by sulphur has been made by Rangaswami and Aldis (19) in their study on the effect of arsenic sulphide on shellac.

PROJECTED WORK

This work on the direct treatment of shellac with sulphur having given some valuable results it is proposed to extend the same to the study on the action of organic and inorganic compounds of sulphur and the examination of the resulting products for their varnish and moulding properties. In this connection mention may be made of the study of such compounds as sulphur chlorides, carbondisulphide, sulphur dioxide, thio-urea and its both aryl and alkyl substituted products, which merit special attention. Some preliminary experiments conducted with the action of sulphur monochloride and thio-urea on shellac varnishes have already given interesting results. Further investigations on the use of these chemicals are being pursued in detail.

In the direct method of sulphurising lac, both the factors of temperature and duration of heat are unduly high. To carry on the reactions more easily, therefore, search is being made for some suitable accelerators or catalysts so that the reaction may be conducted more quickly at lower temperatures. In the investigation of such accelerators, the importance of those which are usually employed in the vulcanising process of rubber has been kept in view. If any suitable catalysts are found, it is proposed to investigate the possibilities of manufacturing bulk samples of the modified shellac in the factory attached to the Institute.

SUMMARY

A study has been made of the effect of sulphur on shellac and its properties. A brief resumé of the work done on similar lines with other natural and synthetic resins and drying oils is given and the possibilities of conducting similar researches on shellac are discussed.

Optimum conditions for the incorporation of sulphur have been determined, and as the result of these investigations it has been found that inclusion of 4-5% and heating at 170°-180°C for nearly half-an-hour show an improvement with regard to varnish and moulding qualities. The sulphurised products are green in colour and evolution of hydrogen sulphide during the reaction indicates some kind of chemical change.

These products have been examined for their varnish and moulding properties and found to possess advantages chiefly in regard to their resistance to water and abrasion, hardness and other mechanical properties.

The moulded products from the sulphurised shellacs have been found to resist 'arc-ing' due to surface carbonisation much better than those made from ordinary shellac, presumably due to the presence of non-conducting sulphur.

The sulphurised shellacs possess another advantage in that they take up any dye stuff in appreciably small quantities giving bright coloured varnishes or mouldings of any desired shade or tint.

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The chemical analysis of these products presented no change in the acid values, but there was a rapid increase of iodine value. The increase in the latter factor seems, however, inexplicable. Further investigations in connection with the action of several organic and inorganic compounds of sulphur, the use of various accelerators during the sulphurising process, from the point of view of improving the properties of shellac have been outlined.

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