

UNITED STATES PATENT OFFICE.

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VEGETABLE DYE AND PROCESS OF MAKING SAME.

1,273,293.

Specification of Letters Patent.

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No Drawing.

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To all whom it may concern:

Be it known that I, HERBERT WARKUP, a citizen of the United States, residing at Kingston, in the county of Ulster and State of New York, have invented certain new and useful Improvements in Vegetable Dyes and Processes of Making Same, of which the following is a specification.

This invention relates to vegetable dyes and processes of making same; and it comprises as a new direct dyeing material vegetable dyeing extracts compounded with a plurality of different mordanting substances, the composition having a neutral or alkaline reaction, being of definite dyeing properties, being capable of directly dyeing cotton and other vegetable fibers of definite shades without the necessity of mordanting and also of dyeing mixed vegetable and animal fibers, such as shoddy, of even and uniform shade throughout; and it also comprises a process of preparing such a dyeing material wherein a vegetable dyeing extract, such as logwood extract, in the presence of at least sufficient water to make it in liquid form, is brought to a boiling heat and a plurality of different mordanting substances, such as for example, copper sulfate and alum or ferrous sulfate and copper sulfate are added and the mixture allowed to stand and cool slowly for a number of hours, say 12 hours, to allow the bases of such mordanting materials to go into "organic combination" and is then treated with a little soda ash or equivalent alkali in amount sufficient to neutralize or render alkaline; and it further comprises as an advantageous embodiment of the described invention the production of standardized direct dyeing vegetable extracts capable of giving definite colors on cotton, linen and other goods by evaporating down a suitable vegetable dyeing extract, such as extract of logwood, quercitron, hypernic, Brazil wood, fustic, sumac, flavin, etc., to a fairly concentrated form, say about 25° Twaddell (16° Be. or 1.125 sp. g.), adding a plurality of different mordanting salts, such as, for instance, sulfate of copper and sulfate of alumina or sulfate of iron, allowing to stand and cool for a number of hours, adding a little soda ash, simmering to get rid of liberated carbon dioxide, boiling several hours, say four hours, in the open and then finally concentrating *in vacuo* to the desired commercial strength of say about

51° Twaddell (30° Be. or 1.155 sp. g.); all as more fully hereinafter set forth and as claimed.

Dyeing with vegetable dyes is of course a very old art, and with care, experience and skill on the part of the dyer results of high excellence may be attained; results which are often difficult of duplication with the synthetic dyestuffs; the so-called anilin dyes. The methods in use however are often somewhat complicated, requiring several steps in the dyeing operation, the use of mordants, etc., so that considerable skill is usually necessary; a fact which has aided, in large measure, in the introduction of such synthetic dyestuffs, and particularly where special shades are required. In general, the dyeing operation is simpler with the synthetic dyes than with the natural. The direct cotton dyes, for example, require no mordanting operation. The synthetic dyestuffs have a definite and constant composition rendering it easy to make dyebaths by rule which will give constant results. This, which was not true of the old fashioned dye baths made by hand by the dyers from small lots of varying materials, is however also substantially true of the modern concentrated dyewood extracts of commerce. Being made in large lots from large lots of material of averaged composition and under chemical control they are also substantially constant in composition and can be used to give readily reproducible dyebaths; dyebaths dyeing exactly the same shades. As with the artificial dyestuffs, it is merely a matter of weighing or measuring the right amounts to make a dyebath of standard composition.

It is the object of the present invention to provide materials which carry the simplification in the use of vegetable dyes a step farther. To this end, a concentrated vegetable dyestuff extract, such as extract of logwood, quercitron, hypernic, fustic, sumac, flavin, etc., is made carrying incorporated in it mordants in such a state of association as will enable the compound material to be used not only for dyeing but for mordanting. As a rule, in dyeing with vegetable extracts mordanting is a separate operation, the cloth, yarn, thread or other material being treated with mordants to fix the dye and then dyed or vice versa. As I have found, however, if the mordants be incorporated in the dyestuff extract the

two operations may be combined in one. I find that in practice in order to do this and make a stable composition of desirable characteristics for the present purposes, it is necessary to incorporate two or more different mordants with the material. As for the reason for this, I am not aware and content myself with noting my observation. It may however be that with the use of two different salts in the composition, double salts of greater stability are produced. Since the different mordants in connection with any dye extract produce widely different results, the choice and character of the mordants will of course depend upon the results to be accomplished. But with the use of two mordants producing together the colors desired, concentrated composite dyewood extracts can be made which with addition of water will give at once a compound bath directly dyeing cotton and other vegetable fibers a definite predetermined shade. And with the present compositions I have noted that the same dyeing effect is also produced on animal fibers such as wool and silk. By the use of the present compositions composite fabrics such as shoddy, can be directly dyed without difference in the shade given the different fibers; a highly desirable result.

I have found that in the best way of performing my process, it is advantageous to evaporate the dyestuff extract *in vacuo* down to a certain concentration less than the final concentration and then add and incorporate my mordanting materials. After the addition and thorough mixing, the material is allowed to stand while still hot for a time to enable, probably, the mordanting materials to go into organic combination. After this is accomplished, I usually make the materials neutral or slightly alkaline with sodium carbonate or analogous alkali, heat to expel carbon dioxide, and effect whatever combinations may occur; and thereafter finish the evaporation *in vacuo* to a standard commercial strength, usually about 51° Twaddell. The first concentration may be, and advantageously is, to about 25° Twaddell. For producing a direct one bath dye with a logwood base, I may use copper sulfate and potash alum or copper acetate and chrome alum, and, in fact, the permutations among mordanting materials allow me to make composite extracts capable of dyeing a very wide variety of colors.

In one specific embodiment of the present invention I extract quercitron bark in any of the known ways and then partially concentrate the extract *in vacuo*; the concentration being carried as far as about 25° Twaddell. To the boiling mixture, I next add 3 parts of copper sulfate and one part of ordinary crystallized iron sulfate. I allow the mixture to stand for about 12 hours,

cooling slowly. It is then reheated and a sufficient amount of soda ash or dry sodium carbonate added to neutralize, and the mixture slowly simmered for a time to get rid of carbon dioxide. The composite mordanting dyestuff extract is then boiled for about four hours in suitable exaporators, (open pans may be used) and is then given a final concentration *in vacuo* to the required strength. The strength given the final product may be such as the market requires. A convenient commercial strength is 51° Twaddell. The compound extract however may be reduced by evaporation to a paste or to a solid, or to the so-called "crystals" or powder.

The particular composition made as just described dyes cotton and other vegetable fibers (and wool and silk as well) in buff shades. For use, it is simply mixed with hot water to give a dye bath of the desired concentration. As a rule, I use about 30 parts of the compound extract made as just described for 100 parts of material to be dyed in a standing bath. Dyeing is very simply performed, it being merely necessary to immerse the material in a dye bath and maintain it wet therewith for a desired period of time, say, in most cases, about an hour. It may then be removed, washed and dried. No further steps are necessary. The color produced is fast to light and washing.

The use of these composite standardized compositions containing both the dyewood extract and the mordants in chemical combination renders dyeing a much simpler operation; the operation in its simplicity being fairly comparable with the ordinary practice of dyeing with the "direct" synthetic cotton dyes.

Direct dyes made under the present invention are concentrated materials containing vegetable coloring matters and mordanting materials, said mordanting materials being, probably, in organic combination. The concentrated material may be of the standard commercial strength of 51° Twaddell or stronger. Evaporation may be conducted so far as to give solid materials, whatever the concentration, the material on dilution with water gives a dye bath capable of dyeing cotton, wool, silk and other fibers, in one operation, with production of fast and permanent colors.

What I claim is:—

1. As a new composition of matter, a dyeing material comprising an extract of vegetable dye material containing a plurality of mordants in organic combination, such bath being neutral or slightly alkaline and being capable of mordanting and dyeing directly cotton goods and the like in the one operation.

2. As a new composition of matter, a dye-

ing material comprising an extract of quercitron containing a plurality of mordants in organic combination, such bath being neutral or slightly alkaline and being capable of mordanting and dyeing directly cotton goods and the like in one operation.

3. A standard direct mordanting and dyeing composite material comprising a vegetable dyeing extract of about 51° Twaddell strength containing a plurality of mordanting materials in organic combination therewith, the material being neutral or slightly alkaline in character and being capable on redilution of producing a dyebath directly dyeing and mordanting cotton and the like in one operation.

4. A standard direct mordanting and dyeing composite material comprising a quercitron extract of about 51° Twaddell strength containing a plurality of mordanting materials in organic combination therewith, the material being neutral or slightly alkaline in character and being capable on redilution of producing a dyebath directly dyeing and mordanting cotton and the like in one operation.

5. The process of making standardized

direct and mordanting vegetable extracts which comprises extracting a vegetable dye material, evaporating the extract to form a thick liquid, treating this thick liquid at a boiling heat with a plurality of mordanting substances, allowing it to stand for a time, neutralizing with sodium carbonate, heating in the open to get rid of carbon dioxide and finally evaporating *in vacuo* to a standard commercial strength.

6. The process of making standardized direct and mordanting vegetable extracts which comprises extracting quercitron, evaporating the extract to form a thick liquid, treating this thick liquid at a boiling heat with a plurality of mordanting substances, allowing it to stand for a time, neutralizing with sodium carbonate, heating in the open to get rid of carbon dioxide and finally evaporating *in vacuo* to a standard commercial strength.

In testimony whereof, I affix my signature hereto.

HERBERT WARKUP.

Witnesses:

E. A. MICHAEL,

CHARLES R. DELANEY.

Copies of this patent may be obtained for five cents each, by addressing the "Commissioner of Patents, Washington, D. C."