

UNITED STATES PATENT OFFICE.

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TO NITRITFABRIK AKTIENGESSELLSCHAFT, OF CÖPENICK, NEAR BERLIN, GERMANY.

PROCESS OF MORDANTING WOOL.

No. 879,553.

Specification of Letters Patent.

Patented Feb. 18, 1908.

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To all whom it may concern:

Be it known that I, SIGMUND KAPFF, a subject of the King of Würtemberg, residing in Aix-la-Chapelle, Prussia, Germany, have
5 invented an Improved Process for Mordanting Wool, of which the following is a specification.

The present invention relates to a process for mordanting wool with aid of reducible
10 metal combinations, especially with those of chrome. For this purpose, lactic acid, lactolin, vegetalin, lignorosene, and tartar have already been employed. These processes labor under the disadvantage that the mor-
15 dant bath is not fully used up, and the reduction of the metal combination in question, for instance, bichromate of soda, always remains more or less incomplete.

The object of the present invention is to do
20 away with this disadvantage, and this is accomplished by employing formic acid as a reducing agent for the mordant, from which there results, as shown by practical use, a complete utilization of the mordant bath, as
25 the reducible metal combination in question, for instance bichromate of soda is reduced to an extraordinary extent (100%). Hence, a great saving can be effected. Formic acid is cheaper than the similar reducing agents for
30 mordants hereinbefore named.

When employing the usual mordants, the reduction of, for example the bichromate generally used for chrome-mordants, is to a great extent incomplete, even when sul-
35 furic acid is simultaneously employed. The addition of sulfuric acid generally, as in lactolin for instance, increases the reducing action, but it increases the cost and causes a loss of time as well, and the working of such process
40 necessitates special attention and skilled workmen. For, if sulfuric acid is added at the wrong time and not with sufficient care, uneven mordanting and dyeing results. The use of other cheap mordants, for instance, bi-
45 chromate and sulfuric acid and oxalic acid, has the disadvantage that the wool suffers ("burns"), and besides the reduction is very deficient.

When formic acid is used as a means of re-
50 duction, as herein described, dyeing experiments with hematoxylin have shown that when mordanting with the use of free formic acid, the chrome is completely reduced and

fixed into the fiber. After mordanting with free formic acid, the bath is completely ex- 55
hausted and as clear as water, while when use is made of lactolin (usually designated as the best mordant or auxiliary mordant) without sulfuric acid, reduction does not take
60 place so completely or even at all. If sulfuric acid is used this acid sets free chromic acid, which is reduced by the reducing agent present. But the sulfuric-acid acts on the wool in an injurious manner. If free formic-
65 acid were present in such bath, the liberated chromic-acid would be reduced at once by the formic-acid, so that the chromic-acid is thoroughly exhausted whether the whole or
70 only a part of the quantity of wool is present which is intended to take up the chromic mordant. I have discovered that by setting free the chromic-acid or by using free chro-
mic-acid the chromic mordant is not suffi-
75 ciently utilized and the action of the same cannot be sufficiently regulated, since the de-oxidation of the chromic compound takes place independent of the wool to be mor-
danted. I have further found that this in-
convenience may be overcome if the use of
80 sulfuric acid or of free chromic-acid or of an agent independently setting free chromic-acid is dispensed with and only a bichromate and free formic acid be used, since, as I have
discovered, in this case the deoxidation of the chromic compound does not take place
85 independent of the wool, but the chromic compound is reduced only in the presence of and apparently in a certain relation to the quantity of wool to be mordanted which is
80 present in the bath. The formic-acid as such does not appear to set free chromic-acid from
bichromate, but in the presence of wool, ap-
parently under the influence of the wool the formic-acid acts on the bichromate directly
95 in the formation of chromic-oxid.

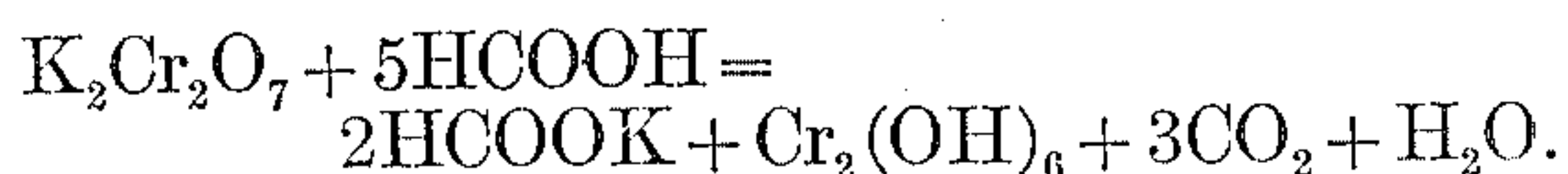
In comparative experiments, it is shown that the employment of about equal parts of bichromate and formic acid results in the most favorable effect, and also that a mor-
dant of 1% bichromate and 1% formic acid 100
is superior to one of 1½% bichromate and 3% lactolin, both in better reducibility and in the full utilization of the chrome, even if an addition of sulfuric acid be made to the
lactolin mordant referred to. The color 105
purity is the same in both cases. The formic

acid offers the further advantage of a very slow action in the mordant bath, so that the chrome is decomposed very uniformly and the dyeings come out very evenly and uniformly. Lactic acid and sulfuric acid act too rapidly and therefore without uniformity; the same can therefore only be used at most for loose wool. Lactolin, without sulfuric acid, does not exhaust the mordant bath. If sulfuric acid is employed for this process, there exists, as in the employment of lactic acid and sulfuric acid, the danger of lack of uniformity in the mordanting and dyeing.

When formic acid is employed, the addition of sulfuric acid, as in the other mordants, is not only unnecessary, but has in fact a deteriorating effect, since it has been shown that when sulfuric acid is also employed, the formic acid evaporates more easily. The formic acid mordant is also more simple than the other mordants, and is better, because of the absence of all noxious ingredients, permitting a continued use of the mordant bath, so that considerable saving in steam, etc. may be effected. After the mordanting in the bath there remains only the entirely indifferent formic potassium, which neither attacks the wool nor interferes with the later spinning process. In consequence, the wool does not need to be rinsed after mordanting, but only cooled off, and can then be dyed at once. By reason of all these advantages the formic acid mordant is better, cheaper, and simpler than all other mordants.

The carrying out of the process may be more clearly illustrated by means of the following example:—100 kilograms of wool are put into a bath which contains about 2000 to 3000 liters of water, 1 kilogram of formic acid and 1 kilogram of bichromate of potassium. In this bath the wool is boiled for one and a half to two hours. After cooling off the wool thus mordanted, the damp material is dyed in the manner intended. So it can be put into a bath which contains 2000 to 3000 liters of water, 20 kilograms of anthracene blue (in dough) and 10% acetic acid. In this the material is dyed in the manner of the alizarin dyeing. Formic acid may be employed, in combination with bichromate, for all mordant dyes. The neutral salts of formic acid cannot be employed for the same purpose, because they remain entirely indifferent. Here, an addition of sulfuric acid would also be of no use, probably for the reason that in the considerable dilution of the sulfuric acid, which comes in question for mordant baths, the formic acid cannot become free. Acid solutions of formates only act in proportion as they contain free formic acid.

In reducing bichromate (according to the present invention) in the presence and under the influence of the fiber and upon the same by free formic-acid at a boiling temperature, the liberating or setting free of chromic acid cannot be observed. It appears that under the influence of wool the chromic-oxid is directly formed by the action of the free formic-acid on the bichromate and that this formation of chromic oxid can only be observed if wool is present. This action of the free formic-acid on the bichromate is apparently caused by the wool present and is therefore automatically regulated by the wool itself, so as to avoid a formation of chromic-oxid not required by the wool. This action may be chemically explained by the following equation:—



This equation indicates that 1 part by weight of bichromate requires 1 part of formic acid (85%). This action of the formic acid was heretofore unknown, especially the fact that this reduction takes place in extremely diluted solution and in a quantitative manner and that the presence of sulfuric acid is harmful and not advantageous, whereas when employed with other reducing agents it acts favorably.

The described action of the free formic-acid on the bichromate in the presence of wool is performed, as described, at boiling temperature thus easily exhausting the bath to an advantageous degree and without injuring in any way the wool, while a bath containing bichromate and sulfuric-acid would severely injure the fiber at the boiling temperature, which therefore is not to be used in the latter case and consequently a sufficient exhaustion of the bath is not possible.

I claim as my invention:

1. The herein described process of mordanting wool, consisting in bringing bichromates and free formic-acid into contact with the wool at a boiling temperature so that they may act upon each other under the influence of the wool without the addition of sulfuric-acid for setting free the chromic-acid.

2. The herein described process of mordanting wool by means of bichromate consisting in bringing the wool into contact with a solution of bichromate and free formic-acid, boiling such solution in the presence of the wool and thus allowing the same to act upon the wool.

3. The herein described process of mordanting wool by means of bichromate, consisting in boiling the wool in an aqueous solution of bichromates and free formic-acid.

4. The herein described process of mordanting wool by means of bichromate consisting in bringing the wool into a bath containing about one part by weight of bichromates and about one part by weight of formic-acid and boiling this bath in the presence of the wool to be mordanted.

In testimony whereof I have signed my name to this specification, in the presence of two subscribing witnesses.

SIGMUND KAPFF.

Witnesses:

HENRY QUADFLIEG,
GERARD PELLERS.