(No Model.)

H. E. SÉRULLAS.

PROCESS OF EXTRACTING AND PURIFYING GUTTA PERCHA.

No. 544,934. Patented Aug. 20, 1895.

FIG. 1.

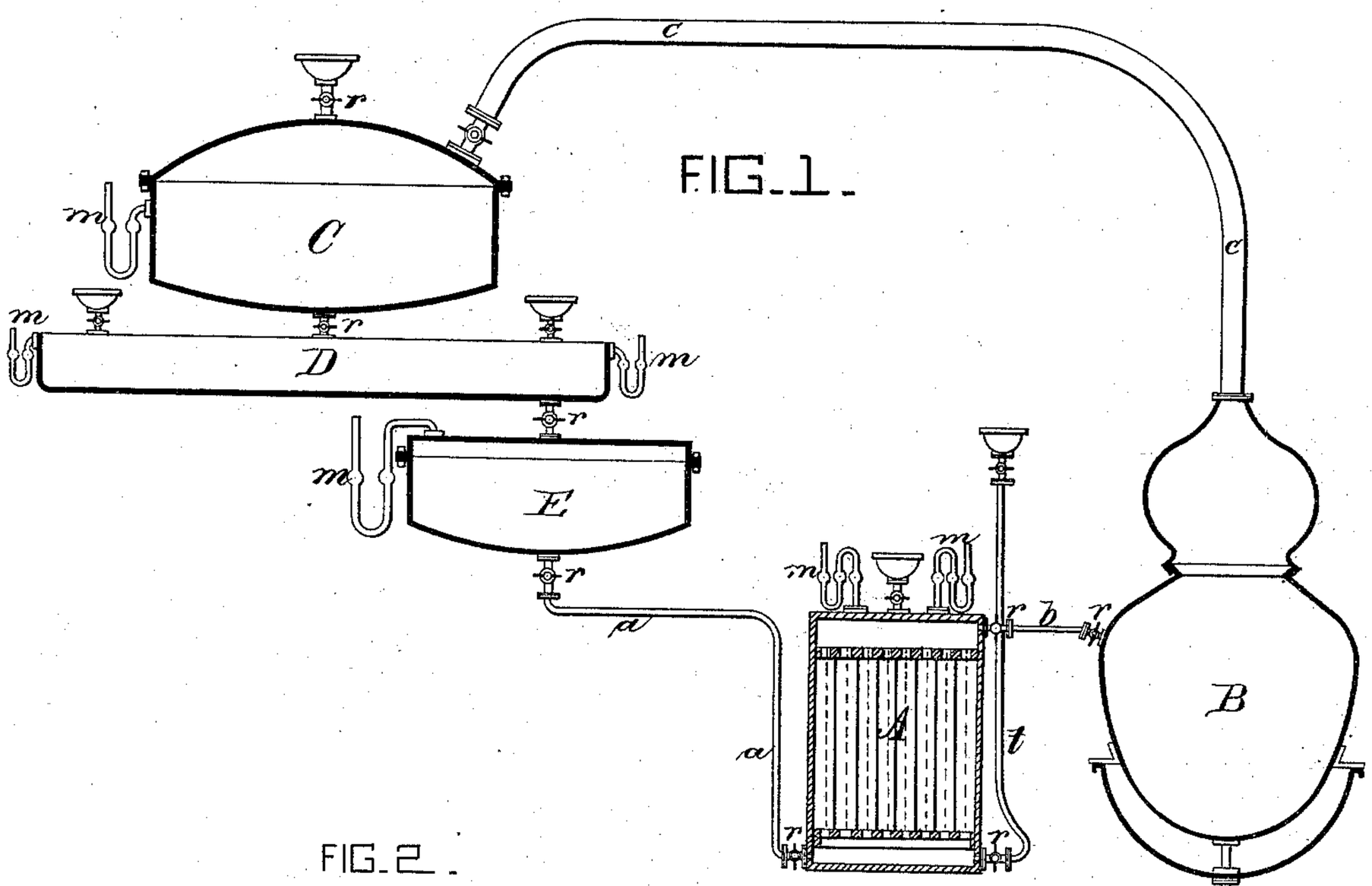
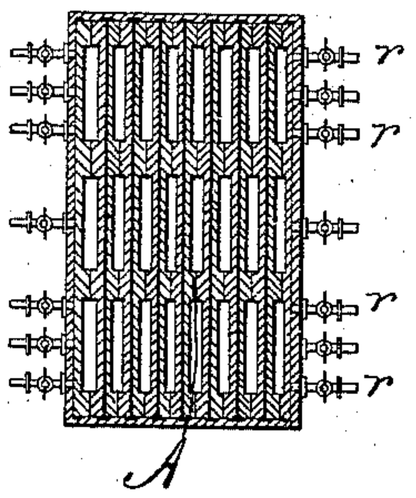


FIG. 2.



WITNESSES:
George Baumann
James Gracin

INVENTOR
Hippolyte E. Sérullas
BY
Howson & Howson
his ATTORNEYS

UNITED STATES PATENT OFFICE.

HIPPOLYTE EUGÈNE SÉRULLAS, OF FORT DE L'EAU, ALGERIA.

PROCESS OF EXTRACTING AND PURIFYING GUTTA-PERCHA.

SPECIFICATION forming part of Letters Patent No. 544,934, dated August 20, 1895.

Application filed March 16, 1893. Serial No. 466,247. (No model.)

To all whom it may concern:

Be it known that I, HIPPOLYTE EUGÈNE SÉRULLAS, a citizen of the Republic of France, residing in Fort de l'Eau, Algeria, have invented an Improvement in the Process for the Extraction and Purification of Gutta-Percha, of which the following is a specification.

My invention has for its object the extraction of gutta-percha from the young shoots or buds and other like parts and, preferably, from the leaves of the isonandra, while preserving absolutely intact the electrical properties of this product—that is to say, obtaining it in a condition suitable for covering the cores of submarine cables. During scientific expeditions in the Malay Archipelago I have learned the proportions of this gum existing in the different parts of the plants of the sapota or sapodilla family and the possibility of substituting very advantageously, in place of the present primitive and wasteful manner of obtaining the gutta-percha, a rational and economical use of the stalks, buds, leaves, and other parts of the isonandra without having to sacrifice the trunk of the trees.

There are obviously only two processes for obtaining the gutta-percha from the leaves and other parts of the isonandra. Either to directly dissolve out only this gum, leaving insoluble all the other substances, or to eliminate these substances successively, leaving the pure gutta as the final residue. The former of these processes destroys the structure of the laticiferous cells. The three hydrocarbons—namely, gutta, fluavil, and alban—forming the constituent parts of these cells in the *Isonandras percha* (Hooker) have each very notable differences of solubility. It results that when they are separated from the solvents they are precipitated in some manner in distinct lots, according to the order of their respective solubility. It then becomes exceedingly difficult, if not impossible, to accurately re-establish their original composition by mixing, grinding, or by any other mechanical means. There is still another objection to this former process. These parts are no longer inclosed in the special cells in which they were found before being dissolved. In place of an aggregation of cells there now only exists a mixture, more or less homogeneous, of a hydrocarbon and two resins. The gum, besides being deprived of all its tenacity before working it up, in which process the Malay natives submit it in warm water, instead of taking a fibrous texture under the rollers after its extraction by the aid of the solvents, in consequence of a considerable elongation of the adhering or consolidated cells, becomes a simple depressed magma of rather a horny appearance, losing more or less rapidly its electric properties, its power of resistance to water under pressure, and the like.

The second process of extracting the gutta-percha from the leaves and other parts of the plant, owing to the preservation of the laticiferous cells, enables the product to retain these last-named properties, which are indispensable in the manufacture of cables. This latter process is that to which my invention relates.

Preliminary operations.—The material under treatment is first dried in a stove in an atmosphere of carbonic-acid gas at a temperature not exceeding 63° centigrade; secondly, pulverized; thirdly, water is drained or washed through at the temperature of the drying process, in order to avoid the coagulation of certain albuminoid matters which an alkaline solution would not remove.

First operation (chemical).—When the washing water from the vegetable powder flows away colorless I pour the powder, along with the water of the last washing, into a digester or closed vessel provided with a valve which will only allow an internal pressure of about eight atmospheres. I cover this saturated powder with about twenty centimeters of water, to which is added potash or soda in the proportion of three parts, by weight, of the alkali to one hundred parts of the powder being treated, or a carbonate of one of these alkalies may be employed containing the base in the same proportion. I keep the contents of the digester at a temperature varying between 106° and 108° centigrade for two hours. Care must be taken that the temperature does not reach 110° centigrade, at which tempera-

ture the fluavil would begin to be injured and decomposed. I then pour the contents both liquid and solid onto a filter, where the residue should be thoroughly washed. I preferably effect this washing by the process known as "filtration by section," and with watts raised to a temperature of about 80° centigrade. The washing is completed when the water comes out of the filter clear and without color and without any action on red litmus-paper.

Second operation (*chemical*).—The residue or settlement thus washed is then poured into an enameled pan, adding sufficient water to cover it with liquid of about five or six centimeters in depth. This water has preferably added to it hydrochloric acid in the proportion of one and a half parts for every hundred parts of the initial weight of the powder, which in the preceding operation has been reduced to nearly three-fourths of its original weight. The pan in which the treatment with acidulated water takes place is provided with a mechanical agitator and a false or double bottom, permitting of heating by steam. When the mixture has been heated for about half an hour at a temperature near the boiling point of water under a pressure of seven hundred and sixty millimeters, I pour out the contents of the pan into a filter, washing it several times on the said filter with warm water, as in the preceding operation. The washing is completed when the water ceases to turn blue litmus-paper red.

Third operation (*mechanical*).—The residue thus obtained is compressed, then spread out under a press at a temperature slightly above 55° centigrade. I then wash it for a little while in running water not exceeding 50° centigrade in temperature. The solid mass thus gets rid of a portion of the interposed cellular or cellulosic powder.

Fourth operation (*mechanical*).—The impure gutta-percha, which forms the solid residue after the mechanical treatment above described, is then triturated in a pug-mill, then passed under rollers, where it is spread out in very thin sheets.

The pug-mill and rollers which I employ may be analogous to those usually employed for the purification of ordinary gutta-percha of commerce.

Fifth operation (*chemical*).—These sheets of gutta-percha thus produced are then stretched on metallic frames which may be arranged to fit in the grooves of a wooden chest lined with metal (zinc or galvanized sheet iron). I introduce these frames into the grooves, which are vertical, so arranged that the space between any two consecutive sheets is from five to six millimeters. I then pass slowly through the said chest a stream of ammonia having in solution precipitated oxide of copper—in other words, a stream of liquid similar to that known in chemistry as "Schweizer's reagent." I continue to pass this liquid until a few cubic centimeters of of the ammoniacal copper liquor cease to give any precipitate of cellulose on the addition of some drops of a diluted acid. A current of carbonic acid or hydrogen gas in a pure and dry condition is then caused to pass between the sheets in the chest, which is hermetically closed during the whole of the operation.

Sixth operation (*mechanical*).—When taken from the frames, the sheets of gutta-percha which, as seen through a magnifying-glass, appear to be full of small holes, are placed in a drying pug-mill similar to those usually employed in the manufacture of gutta-percha. They are there hardened and agglutinated, after which it only remains to roll them afresh or, better still, to compress them into cakes for better preservation.

In order to enable the treatment described under fifth heading to be better understood and what is essential, I will now briefly describe the apparatus which is indispensable for obtaining the complete elimination of the cellulose, and that without injuring in any degree the properties of the gutta-percha.

In the accompanying drawings, Figure 1 is a view showing the different vessels in section in their different positions. Fig. 2 is a sectional plan of the chest A.

A is the chest or washer, which contains the metal frames over which are stretched the sheets of gutta-percha. The ammoniacal copper liquor enters by the pipe *a* and leaves by the pipe *b*.

t is a glass-gage tube with metal socket and funnel.

B is the still, heated by steam or water bath, whose goose-necked pipe *c* brings back in solution in the water recovered ammoniacal gas. C is the reservoir containing the ammoniacal water; D, a small barrel or cylinder having an internal agitator or rouser of any known kind (paddles, for example) and in which the solution of the oxide of copper is effected.

E is the receiver for the copper ammoniacal liquor, whence it flows into the chest A.

m are safety-tubes, and *r* cocks for working the apparatus.

One chest A is preferably fed by several sets of apparatus B, C, D, and E.

I claim as my invention—

1. A process of extracting gutta percha consisting in treating the parts of the tree with an alkali or its carbonate, then treating this residue with dilute hydrochloric acid, at the same time triturating it, then spreading this last residue into sheets and then treating the sheets to a stream of ammoniacal copper liquid, substantially as set forth.

2. A process of extracting gutta percha con-

sisting in treating the parts of the tree with an alkali or its carbonates then treating the residue with dilute sulphuric acid, next spreading this last residue out into sheets and treating the sheets first with a stream of ammoniacal copper liquid and afterward with a current of carbonic gas or hydrogen gas, substantially as set forth.

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

HIPPOLYTE EUGÈNE SÉRULLAS.

Witnesses:
FÉLIX HOURAND,
CHARLES THEODORE GRELLET.