

(No Model.)

E. WATEL.
PROCESS OF EXTRACTING PERFUME ESSENCES.
No. 498,830. Patented June 6, 1893.

Fig. 1.

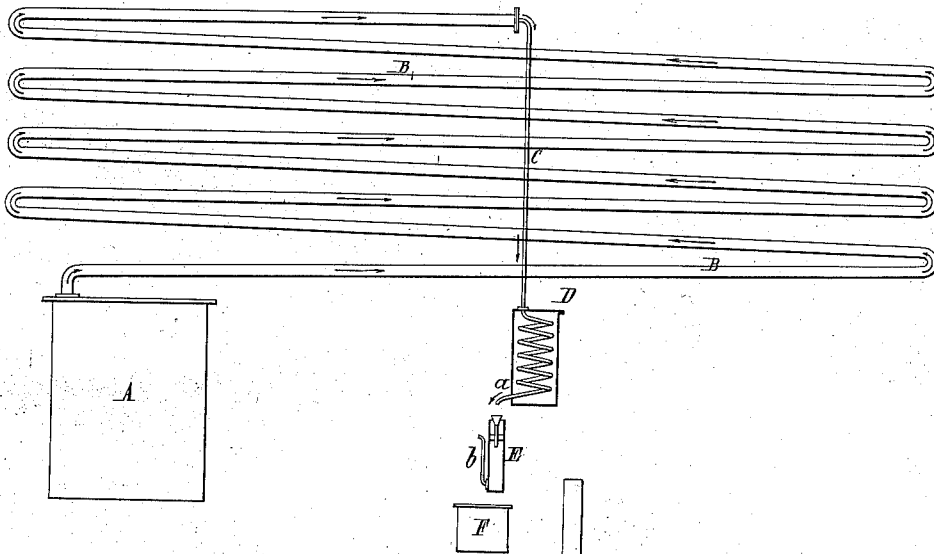
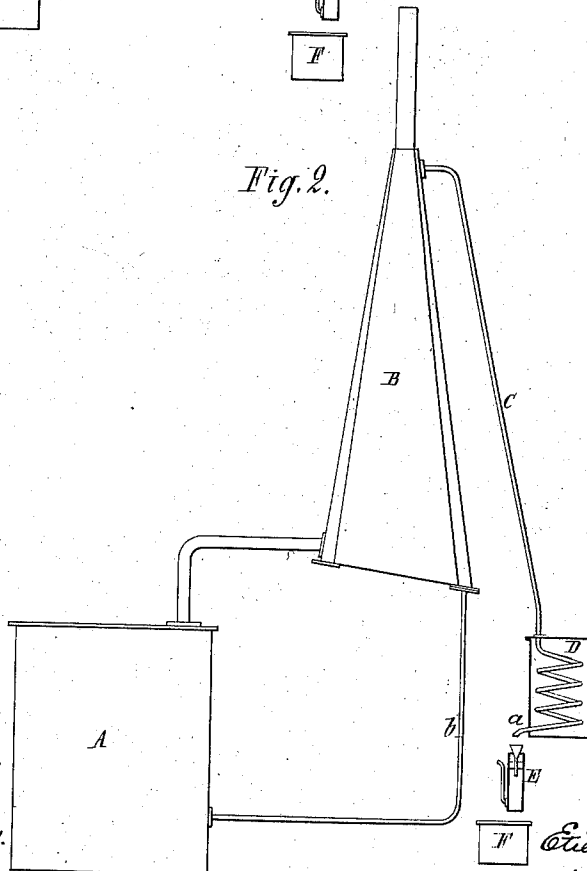


Fig. 2.



Witnesses:
Edouard
B. S. Brumfiel.

Inventor:
Etienne Watel
By *Richardson*
Attorneys.

UNITED STATES PATENT OFFICE.

ETIENNE WATEL, OF PARIS, FRANCE, ASSIGNOR TO THE SOCIÉTÉ ANONYME
DES PARFUMS NATURELS DE CANNES, OF SAME PLACE.

PROCESS OF EXTRACTING PERFUME ESSENCES.

SPECIFICATION forming part of Letters Patent No. 498,830, dated June 6, 1893.

Application filed October 23, 1890. Serial No. 369,127. (No specimens.) Patented in England May 8, 1886, No. 6,259.

To all whom it may concern:

Be it known that I, ETIENNE WATEL, a citizen of France, residing at the city of Paris, in the Republic of France, have invented certain new and useful Improvements in Processes of Extracting Perfume Essences; and I do hereby declare the following to be a full, clear, and exact description of the same, and for which Letters Patent of Great Britain, No. 6,259, dated May 8, 1886, were granted as a communication from my assignee, La Société Anonyme des Parfums Naturels de Cannes.

The present invention relates to the extraction of perfume essences, or odoriferous essential oils, from vegetable matters, such as orange-blossoms, rose, jessamine, geranium, lavender, mint, clove, &c., and consists in an improved process, whereby the essential oil is obtained in a liquid state comparatively free from water of condensation.

The process is carried out as follows:—The vegetable matter to be treated is heated with water in an alembic and the watery vapor, together with the vapors of essential oils passing off, are received in an ascending condenser, which allows the watery particles as they condense, to return to the alembic, while the vapors of the essential oil, which is propelled upward by a column of aqueous vapor strictly sufficient for the purpose, rise to the top of the condenser, where the mixture of vapors meets with a suitable known refrigerating material, which condenses both the watery vapor and that of the essential oil, thus producing the latter in a liquid state with a small admixture of water. The proportion of water used and the condition of the vegetable substance when introduced into the alembic, are the same as in ordinary processes.

The essential difference between this process and that usually employed is, that in the latter case the whole of the aqueous and essential oil vapors are condensed together, so that the latter are disseminated in a large quantity of water; while according to the present invention the greater part of the aqueous vapor is condensed separately from the essential oil and is returned to the still, only as much water being carried along with the volatile oil as is strictly necessary to convey this forward to the condensing medium.

From this small amount of water the essence can be afterward very readily separated.

The process is based upon the laws governing the tension of vapors, and on the different relative solubilities of the vapors or essential oils in steam. Take for example the distillation of orange blossoms: under the influence of ebullition in an alembic, or still, there is produced a mixture of steam and vapor of essential oil, which mixture rises in the ascending column of the condenser. It would be supposed that the vapor of the oil, because its point of condensation is higher than that of steam, would be first to condense and return to the still, and that at the top of the apparatus nothing but steam would arrive. But such is not the case. It is the steam that in great part condenses, while all the vapor of the oil reaches the top of the column, mixed with the uncondensed portion of the steam. This is due to the fact that the weight of the vapor contained in the determined volume of mixture is very small, as compared with that of steam alone; and to the operation of the law above referred to, namely "the tension of a vapor in a mixture is equal to that which it would have if it occupied alone the space occupied by the mixture," it follows that the tension of the vapor is also very slight, compared with that of the steam; or in other words, the condition of the vapor is the same as if it were submitted to a strong vacuum. Obviously under these conditions its point of ebullition, or inversely of condensation, is much lower than that of steam (100° centigrade). The state of tension of the vapor may be approximately determined by considering the fact that the essence-oil contained in the orange blossom represents 0.0015 of the weight of the blossom, and that the steam formed during ordinary distillation (which will be found condensed in the form of orange blossom water in the cooled serpentine) is equal in weight to that of the treated flower. That is to say, supposing (to simplify matters) that the vapor of the essential oil is of equal density to steam, the weight of the vapor in a determinate volume of the mixture is only 0.0015 of the steam. Under these conditions, the ratio of tension of the vapor is to that of the steam as one to six

hundred, or thereabout. This vapor, therefore, is far from its point of saturation or condensation under the conditions of temperature and pressure existing during the operation; while the steam is very near that point; and it will be understood that when such a mixture traverses a long ascending column the steam would have a tendency to condense much more rapidly than the vapor, so that the proportion of vapor in the mixture increases progressively as the mixture rises in the column until it reaches such a proportion that the tension of this vapor corresponds precisely with its point of condensation under the conditions of temperature and pressure existing. If we consider also that most of the vapors of the essential oils are but little soluble in steam, it will be seen that the proportionate quantity of vapor can greatly exceed that which would result if the law of tension alone were considered. Consequently, it is possible to collect on top of the column, a mixture in which the proportion of steam is materially reduced with respect to that actually produced, and may be collected entirely in the cold condenser coil, by the ordinary method. In fact, I collect about from eighty to one hundred liters of condensed water from one thousand kilograms of orange blossoms, instead of one thousand liters, which would be collected by the ordinary method.

The above described process can be carried out in any suitable construction of distilling apparatus composed of the before described parts.

By way of example I will describe the construction employed by preference referring to the accompanying drawings.

The apparatus shown in Fig. 1, consists of an alembic A above which is an ascending condenser formed of nearly horizontal pipes B connected at the ends by bends, so as to afford a considerable extent of travel for the vapors circulating therein. The end of the top pipe communicates by a pipe C with a

refrigerating coil D where the condensation is effected. The aqueous vapor that condenses in the tubes B flows back in the alembic, while the uncondensed vapor of perfumed essence flows on to the condenser D together with a very small proportion of uncondensed watery vapor, and flows off in the condensed condition through the spout *a* into a recipient E, in which the essence separates by gravitation from the water, which sinks to the bottom and flows off through the bent tube *b* into a vessel F.

In the apparatus shown in Fig. 2, the ascending condenser B consists of a double conical vessel with slightly inclined sides, in which the water condenses and collects at bottom flowing back into the alembic by a pipe *b* while the essence vapors together with any uncondensed watery vapor are conducted through pipe C to the refrigerating condenser D as before.

Having thus described my invention, what I claim is—

The process of extracting from vegetable substances the odoriferous essential oils less volatile than water and but slightly soluble therein, which process consists in heating the vegetable substance with water in an alembic, producing a mixture of steam and vapor of the essential oil of such substance the steam being greatly in excess, causing the mixture to pass into an ascending column, wherein the greater portion of the steam is condensed and returned to the alembic, and finally condensing the mixture of vapor of the essential oil and uncondensed steam which reaches the top of the column, substantially as described.

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

ETIENNE WATEL.

Witnesses:

R. J. PRESTON,
JOSEPH COURNIER.