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STUDY OF THE DESORPTION OF ETHYLENE OXIDE FIXED ON VARIOUS MATERIALS DURING STERILIZATION BY A NEW PROCEDURE

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16. Abstract

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STUDY OF THE DESORPTION OF EHTYLENE OXIDE FIXED ON VARIOUS MATERIALS DURING STERILIZATION BY A NEW PROCEDURE.

M. Lacomme, M. Chaigneau and G. Le Moan

Summary /91*

A new continuous sterilization process using ethylene oxide has been studied in comparison with a classical method in order to evaluate gas retention as a function of time and temperature on polyethylene, PVC and rubber materials.

Sterilization methods using ethylene oxide can be classified into two large categories:

The first uses ethylene oxide mixed with air generally in a proportion of 50%. This technique has the advantage of being economical. Its major drawback is the danger of explosion. Under a pressure of one atmosphere the ethylene oxide-air mixture is explosive for volume-to-volume concentrations of 3-80%. The danger of explosion is limited by using an armored, watertight sterilization chamber under strict operating conditions. A vacuum of a few millimeters is created in the chamber, then the gas is introduced in suitable doses, but so as to preserve a partial vacuum in the chamber, thus preventing any possible diffusion of the gas to the exterior. The sterilization time is a function of the material to be sterilized, the temperature and the ambient humidity. At the end of sterilization the ethylene oxide is removed by pumping followed by one or several rinsings with filtered air.

The second classic method uses ethylene oxide mixed with different gases. The most common are carbon dioxide, nitrogen and freons. The advantage of these mixtures is that they

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* Numbers in the margin indicate the pagination in the foreign text.

remove the danger of explosion. The concentration of ethylene oxide generally used is 10-20% by volume so as to obtain a concentration greater than 400 mg of ethylene oxide per cubic meter. The sterilization is done in armored containers. After first putting the interior of the chamber under a vacuum the gaseous mixture is generally introduced at a pressure greater than atmospheric pressure (2-3 atmospheres) in order to reduce the contact time which would be longer taking into account the small concentration of ethylene oxide in the mixture. This poses safety problems which are not insignificant and requires the use of large quantities of gas which increases the cost of production. At the end of sterilization one or several rinsings are performed with sterilized air. /92

These two classes of techniques have the following in common: the sterilization process is discontinuous, it uses large containers and requires significant safety precautions and trained and experienced personnel.

A new original technique has been developed. It consists of injecting a gaseous mixture consisting of 12% ethylene oxide in a "freon" into a pocket of plastic material containing the material to be sterilized. The pocket constitutes the sterilization chamber allows the ethylene oxide to diffuse slowly. It is placed in a ventilated chamber heated to 50°C where sterilization and desorption take place simultaneously. This process which we will call S[1] permits continuous sterilization. It does not require a large container. The procedure is simple and quick. Therefore it seemed interesting to us to study the desorption rate of ethylene oxide fixed on plastic materials by this process in comparison

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1. Distributed in France by the Élis group under the name of Stérijet.

with a process M[1] largely in use today.

Materials and Method

1. Materials

Three types of materials were tested:

- a low pressure, high density 6050 polyethylene with very few additives and in the form of irregularly shaped "caramels" 2 mm long and 3.8-4 mm in diameter.
- two PVC's: BTS 140 A7 lucolene parallel:pipedal "caramels" (4 x 3 x mm) containing 68% PVC and 2312 B PVC, also in the form of "caramels" containing 56.4% PVC.
- a latex rubber of unknown composition used for making Foley tubes with an i.d. of 4 mm and an o.d. of 8 mm.

All of these materials are used in the manufacture of medicosurgical products. Their composition is fully known, with the exception of the latex, and it will be dealt with in future monographs of the French Pharmacopia.

2. Apparatus

We compared process S with process M. In process S the objects to be sterilized, impregnated with ambient humidity (40-50%), are put singly or in groups into a pocket of complex and specially studied plastic material which takes the place of the sterilization chamber. This pocket is made, on the one hand, of polyamide, which is impermeable to ethylene oxide, and, on the other hand, of low pressure polyethylene, which is permeable to ethylene oxide. It is impermeable to air, but allows the sterilizing gas to diffuse slowly to the outside through the polyethylene by means of a window made in the polyamide layer. The opening of the pocket is first of

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1. Mallet apparatus.

all connected to the device by means of which it can first be emptied of air -- this is confirmed by the complete collapse of the walls -- and secondly the device injects a premeasured dose of a gaseous mixture containing 12% ethylene oxide and 88% trichlorofluromethane. The pocket is then heat sealed and immediately placed in a ventilated and heated chamber. All of these operations take only a few seconds, Then, con- /93
comitant with this begins the sterilization, or more precisely the desorption. During the first hours the concentration at a temperature of 50°C is greater than the minimum dose of bactericide. This length of time corresponds to an effective sterilization time. Further, desorption continues until the ethylene oxide has been removed.

Process M uses a mixture of ethylene oxide of air in a proportion of about 50%. It is characterized by sterilization at 40°C at a pressure less than atmospheric pressure. The steps involved in the sterilization process are as follows:

1. Packets containing the objects to be sterilized are placed in the container.

2. A vacuum is created by a turbine pump in order to remove the air from the container.

3. Sterilizing gas is introduced into the chamber at a pressure ranging between 600 and 650 mm of Hg.

4. Contact continues for six hours.

5. Removal of ethylene oxide by creating an initial vacuum followed by introduction of air until atmospheric pressure is released, followed in turn by a second (rinsing) vacuum and a final injection of air.

The packets are then placed at the outlet of the device in the desorption chamber used in process S.

Experimental Procedure

The tests were carried out identically for all the materials according to the following procedure:

Ten g of plastic materials were placed in packets made of paper on one side and polyethylene on the other and sealed closed. In process S these packets are put into special pockets described above furnished by the manufacturer. The entire unit in this case constitutes a double wrapping which differentiates it from process M, since in the latter process the packets are placed as is into the chamber.

Analytic Method Used

Measure of the residual ethylene oxide in the materials was done according to a method which we have described previously [2]. Measurements were made at 48, 72, 120 and 168 hours after the beginning of sterilization. The time lag between the two processes is due to the fact that in process S the handling operations last only a few seconds, whereas in process M they take at least six hours.

Results

The results obtained are given in Tables I, II, III and IV.

Table I. Process S. Ethylene oxide concentration in ppm in polyethylene (PE), lucolene PVC and in PVC 2312 as a function of the aeration time at 50°C.

Aeration time in hours	PE	PVC Lucolene	PVC 2312 B
48	5	57	31
78	2	14	12
120	< 1	3,3	2,2
168	< 2	< 2	< 2

Table II. Process M. Ethylene oxide concentration in ppm in lucolene PVC and PVC 2312 as a function of aeration time at 50°C

Aeration time in hours	PVC Lucolene	PVC 2312 B
48	368	< 50
72	17	2
120	1,2	1,7
168	< 2	< 2

Table III. Process S. Ethylene oxide concentration in ppm in a latex rubber as a function of aeration time at 50°C

Aeration time in hours	Rubber
24	10
48	2,2
96	< 2

Table IV. Process S. Ethylene oxide concentration in ppm in polyethylene (PE), lucolene PVC and in 2312 PVC as a function of aeration time at ambient temperature.

Aeration time in days	PE	PVC Lucolene	PVC 2312 B
2	459	7 265	6 277
3	318	2 981	2 963
7	186	2 489	1 269
14	64	444	75

The following facts emerge from these various results:

1. Polyethylene retains less ethylene oxide than PVC, as we have stated previously [1].

2. After 48 hours the concentration is lower with process S than with process M. This would be due to the difference in ethylene oxide concentration in the sterilizing mixture.

3. Desorption is slightly more rapid with process M. This would be explained by the fact that in order for the ethylene oxide to be removed it must pass through wrapping layers in process S instead of one. The first ordinary wrapping does not retain any ethylene oxide and the second only allows the gas to diffuse slowly. However, the increase in the storage time with process S is only a few hours with respect to process M (Tables I and II).

4. The desorption chamber is essential with process S. The figures obtained with desorption at ambient temperature in fact very high (greater than 50 ppm after 14 days). Nevertheless, it also turns out to be desirable to use the desorption chamber in process M (Table IV). In fact, by comparing these results to findings obtained for identical materials under similar conditions [3] it would appear that the storage time is reduced from 15 to 5 days with aeration at 50°C.

5. Ethylene oxide penetration was checked chemically by means of test papers furnished by the manufacturer of the device which were placed in the packet. However, the gas-object contact time is difficult to determine in process S. According to the manufacturer this time is three hours. Bacteriological checks were done at the Institut Pasteur in

Lille. The results of these tests were negative. The bacteriological effectiveness of this process has also already been studied in the United States by Anderson [4].

6. Even if the desorption results are similar in the two processes, it can be said that process S permits numerous continuous sterilizations (1,000 pockets per day) and moreover at the end of desorption the absence of free gases in each pocket is confirmed by the fact that the pocket is collapsed completely with both sides touching one another. This makes it possible to check that sterilizations are maintained and thus that the wrapping remains intact until the pocket is used.

Conclusion

This original device appears easy to use. The handling time is very short and it permits continuous sterilization. As in most processes using ethylene oxide, the desorption oven is essential and under these conditions the storage times are equivalent to those for processes currently being used.

Summary

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Study of the desorption of ethylene oxide fixed on various materials during sterilization by a new procedure.

by M. Lacomme, M. Chaigneau and G. Le Moan

A new continuous sterilization process using ethylene oxide has been studied in comparison with a classical method in order to evaluate gas retention as a function of time and temperature on polyehtylene.

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