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[54] **PACKAGE COMPRISING PAPER CONTAINING A FORMALDEHYDE RELEASING THERMOSETTING RESIN**
2 Claims, 1 Drawing Fig.

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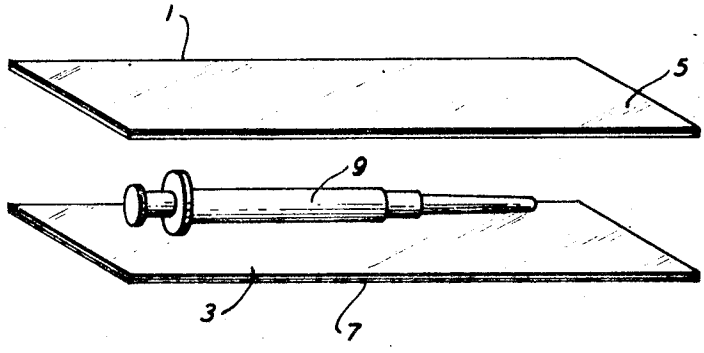
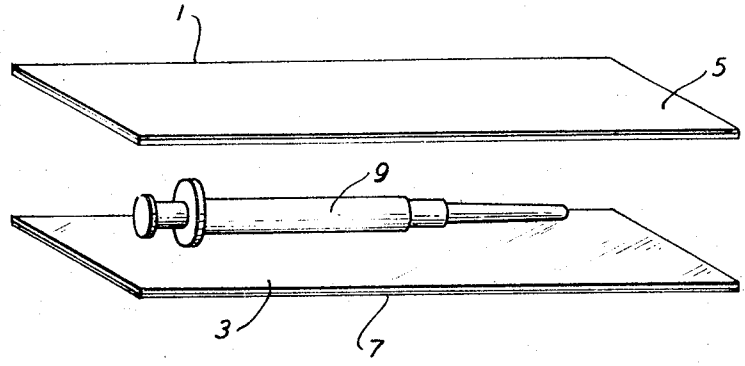
ABSTRACT: A package having a wall, or sheet contained therein, impregnated or coated with a formaldehyde containing uncured resin having pH in the range of 4–9 so that formaldehyde is gradually released upon cure to sterilized articles within the package.





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PACKAGE COMPRISING PAPER CONTAINING A FORMALDEHYDE RELEASING THERMOSETTING RESIN

BACKGROUND OF THE INVENTION

This invention relates to a package useful in packaging and sterilization of disposable surgical instruments. Commonly used methods of sterilization of surgical packages in practice today include long storage and exposure times to ethylene oxide in gas chambers, or exposure to beta ray radiation, or a combination of the two. Time of exposure and handling are a specific restriction of ethylene oxide. Radiation sterilization is rapid but not thought to be as efficient as ethylene oxide. Present packaging material ranges from hard polyethylene capsules to paper heat-sealed and coated with polyethylene or polypropylene.

SUMMARY OF THE INVENTION

Briefly, a paper is coated or has internally incorporated therein an uncured formaldehyde containing thermosetting resin. The resin does not contain free formaldehyde but many $-CH_2OH$ groups which are capable of splitting off as free formaldehyde on heating and/or aging: the reaction is catalyzed by acids or bases. Since it is desired that formaldehyde be released over extended periods such catalyzation is undesireable and accordingly, the pH of the sheet is adjusted to a range between 4 and 9.

This paper is then formed into a package with a surgical instrument placed therein. The sealed package is then subjected to a heat treatment so as to release the formaldehyde. The heat treatment releases sufficient formaldehyde so as to kill the bacteria in the package but it is so controlled as to not completely free all the formaldehyde present. The resin continues to gradually cure with aging due to aforementioned pH control so that the formaldehyde remaining linked with the resin is released continuously until the package is broken for use purposes.

It is desired that the formaldehyde be retained within the package until the latter is broken open. In order to assure accomplishing this, a vapor-proof coating such as polyethylene, is placed on the side of the sheet which forms the outside of the package so as to seal the sheet and prevent release of the gas therethrough.

The formaldehyde containing paper need not be made into a package. For example, the paper can be incorporated within an aluminum foil package, with the paper serving to guard or encompass the surgical instrument so as to inhibit the penetration of the surgical instrument into the aluminum foil, while simultaneously sterilizing the interior of the package.

DRAWINGS

The FIGURE is a schematic diagram showing the parts broken away, illustrating a package made in accordance with the present invention.

DETAILED DESCRIPTION

Any formaldehyde containing thermosetting resin such as phenol formaldehyde, urea formaldehyde, melamine formaldehyde, etc., which is uncured and capable of releasing formaldehyde upon subsequent polymerization may be employed. In the uncured state, these resins are or contain monomers which are mono, di and tri methylol compounds with phenol, urea and melamine. There monomers condense to give ether linkages which decompose to methylene linkages with subsequent loss of formaldehyde.

The resin need not be completely uncured. If, e.g., the resin is 80 percent cured, there is still some formaldehyde available for liberation upon heating or aging. If such a partially cured resin was employed in sufficient quantity, applicant's purpose would be served. Hence reference herein, both in the specification and in the claims, to an uncured resin is meant to include such partially cured resin provided such resins are capable of liberating formaldehyde upon subsequent aging and heating.

The amount of resin incorporated in or on the supporting sheet (which will subsequently constitute an insert or wall of the package) will depend upon the state of cure of the resin. In addition, the amount of resin required will vary depending on the needs of a particular application. In view of these variables, the amount of resin can be varied over a wide range from 0.5 percent to 90 percent by weight of the supporting sheet.

The formaldehyde containing uncured resin may be incorporated internally into a sheet of paper by addition during paper manufacture or it may be coated onto a sheet by any conventional coating operation such as reverse roll, wire rod, air knife, etc.

As hereinbefore noted, the resin will cure and release formaldehyde as a result of aging and heat. An essential control however, is to keep the sheet within a pH range of 4–9. Within this range, cure is slowed down considerably upon aging and the curing process will last an almost indefinite period of time thus assuring formaldehyde release until the package is opened.

In order to maintain the sheet within the aforenoted pH range, the pH is adjusted by the addition of caustic to a pH of 7 at the beater stage or head box prior to paper formation. In addition, a buffer is added at the size press so as to maintain the neutral condition of the finished product. Paper has inherent acidity due to the fiber and it is quite difficult to fully neutralize this by caustic addition at beater or head box stage.

The paper used may be of conventional type used in packaging surgical instruments, such as those made from bleached sulphite and other bleached pulps which impart the required strength and porosity requirements to such a sheet.

In making the paper on the paper machine the following procedure is followed:

1. The pulp is beaten and refined, ready for deposit on the wire of a conventional paper machine.

2. As the pulp is passing into the head box prior to deposition on the wire screen, a highly dilute (not greater than 10 percent) solution of uncured formaldehyde containing resin is mixed therewith. In higher concentrations, the resin will cure. In order to neutralize the acidity of the hydrochloric acid incorporated in the melamine formaldehyde, solium hydroxide is added simultaneously therewith so as to adjust the pH to about 7. Based upon the amount of fiber present, formaldehyde containing resin is added so as to provide a final retention of 1 percent to 10 percent in the final product. This range of final retention is not at all critical but set forth merely as an economically feasible and commercially practical range.

3. At the size-press of the paper machine, a buffering agent is added to the sheet so as to maintain the neutral condition of the finished product. The amount of buffer should be 0.2 percent to 0.5 percent based on the weight of fiber. The paper has inherent acidity due to the alum on the fiber and it is impossible to fully neutralize this during the caustic addition step at the head box. The buffering agent serves to neutralize any free acid radicals that might be present and were not neutralized during the addition of the caustic at the head box. Conventional paper making buffers can be employed such as hydrated sodium borate, potassium-hydrogen-phosphate, acetates, etc.

4. After formation of the sheet on the wire, it is then passed over conventional drying drums and calendered according to conventional practice.

As hereinbefore described, the resin may alternatively be applied by any conventional coating operation such as reverse roll, wire rod, air knife, etc. The resin is applied in aqueous or solvent system depending on the particular resin used. The resin is applied in a highly dilute solution, generally not greater than 10 percent concentration so as to inhibit cure. The resin is applied in sufficient quantities so as to meet the requirements of the particular intended end use.

In choosing whether to coat the resin onto the sheet or to incorporate the resin internally within the paper during the paper manufacturing step, consideration has to be given to the

drying temperatures of the coating apparatus or the paper machine. There must be a minimum amount of a formaldehyde present in order to provide the required kill of the bacteria present in the package and there is less formaldehyde available as the cure of the resin progresses. Generally, the temperatures should be adjusted if possible so that the resin is no more than 90 percent cured.

The resin impregnated paper as above prepared can be incorporated within a vapor-proof package, such as an aluminum foil package, and subjected to controlled heating so as to hasten cure, and release sufficient formaldehyde to kill bacteria present in the package.

If the paper is to be formed into a package, the surface of the sheet adapted to be on the outside of the package is provided with a vapor-proof coating. The coating may comprise any resin which has a low-vapor transmission level such as polyethylene, polypropylene, polyvinylidene chloride, etc. In the alternative, a hot melt coating may be applied comprised, e.g., of a high molecular weight ethylene-vinyl acetate copolymer combined with petroleum waxes.

Referring to the drawing, a package is prepared in accordance with the present invention with sheets **1** and **3**, placed in face to face relation. Sheets **1** and **3** have an uncured formaldehyde containing thermosetting resin incorporated therein by the procedure above described. Sheets **1** and **3** are assembled by heat sealing. The outer surfaces **5** and **7** of the sheets **1** and **3** have a vapor-proof coating so as to seal the formaldehyde within the package. An instrument **9** is placed on top of sheet **3** and a heat seal die is brought into contact with the package causing the edges of sheets **1** and **3** to fuse together.

After assembly, the package is then placed in a heated chamber for 1 hour to several days at a temperature between 100°–200° F. Formaldehyde is released as the resin cures and it is retained in the package by virtue of the vapor-proof coating.

The following are specific examples of papers prepared for use in making packages in accordance with the present invention.

EXAMPLE I

A sheet of paper having a basis weight of 45 lbs. per ream and made of a pulp furnish comprising a mixture of bleached softwoods and hardwoods was impregnated with a 6 percent aqueous solution of trimethylol melamine (monomer which when polymerized forms melamine formaldehyde). The solution was applied through a laboratory size press at room temperature and then dried in a hot air oven at a temperature of 100° F. The dried sheet contained 5 percent of melamine formaldehyde on a dry fiber basis and had a pH of 4.

EXAMPLE II

The same procedure was followed as in example I except that the impregnating conditions were so adjusted that the sheet contained 3.5 percent of melamine formaldehyde on a dry fiber basis. In addition, before drying, the sheet was impregnated with a buffer solution comprising hydrated solium borate so as to adjust the pH of the sheet to 7.

EXAMPLE III

The same procedure was followed as in example I except that the impregnating conditions were so adjusted that the sheet contained 3 percent of melamine formaldehyde on a dry fiber basis. In addition, a vapor-proof coating was then applied to one surface of the sheet comprised of water resistant wax emulsion.

Twelve packages were then formed from each of the papers described in examples 1–3. Prior to edge sealing of the packages, a surgical instrument precontaminated with 10^5 spores of Bacillus stearothermophylus was placed within each package.

The packages were then placed in a hot air oven at 140° F. for period of 1,3 or 7 days as indicated by the Days Contact Time in the table below. At the end of the contact time the packages were broke open and the instruments even incubated in agar for a period of 7 days. A positive indication as shown in the table below means that there was bacteria growth evident after the contact and incubation period.

	Days Contact Time	Number Of Samples	Positives
Example I (5% MFR, pH4)	1	4	4
	3	4	2
	7	4	1
Example II (3.5% MFR, pH 7)	1	4	3
	3	4	0
	7	4	0
Example III (3% MFR, pH4, Vapor Proof Coated)	1	4	0
	3	4	0
	7	4	0

It will be seen from the above results that a package constructed in accordance with the present invention functions to effectively destroy bacteria with a surgical instrument containing package. It will also be noted that pH adjustment to the neutral range and the application of a vapor-proof coating substantially increases the kill.

As will be now apparent, the package herein described affords a convenient and expeditious mode of delivering a sterilizing substance to articles contained therein over extended periods of time with significantly satisfactory results. It is to be understood that the invention is not limited to specific materials and procedures hereinabove described, but may be carried out in other ways without departing from its spirit.

What is claimed is:

1. A package, said package having paper wall means comprising a thermosetting resin which is no more than 90 percent cured having a substantial quantity of $-CH_2OH$ groups capable of releasing free formaldehyde on heating, said wall means having a pH in the range of 4 to 9, the exterior surface of said package having a vapor transmission proof coating thereon, said resin being present in an amount ranging from 0.5 to 90 percent by weight of said wall means.

2. A package, said package containing an article, a sheet of paper material being wrapped about said article, said sheet of paper material containing a thermosetting resin which is no more than 90 percent cured comprising a substantial quantity of $-CH_2OH$ groups capable of releasing free formaldehyde on heating, said sheet of material having a pH in the range of 4 to 9, said package having wall means, said wall means having a vapor transmission proof coating thereon, said resin being present in an amount ranging from 0.5 to 90 percent by weight of said wall means.

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