

The preservation of wet wipes

Moistened towelettes were introduced nearly 50 years ago as a quick way to clean hands after a take away meal. Although the moistened towelette had been around for some time, the development of baby wipes in the 1970s saw an expansion of the market while many more applications started around the mid 1990s. Since then the global wipes market has grown to in excess of \$10 billion in 2009 and is expected to exceed \$13 billion by 2014. Personal care wipes led the market for many years (with baby wipes being the largest sector) but household wipes dominated by 2005 accounting for 45% of sales in North America.¹

Pet care wipes have also entered the market, such as Pawtizer, an antibacterial paw wipe because 'there are as many germs on paws as on human hands'.²

The manufacture of wet wipes is specialised requiring bulky equipment for the manufacturing of the wipe substrate, its subsequent dosing with the 'wet' phase and final packaging. The formulating of the wet wipe solutions may be conducted in-house or contracted out but the application to the substrate for personal care and household products is generally contracted out to manufacturers with the experience and equipment to handle the work.

The main areas of use for wet wipes are set out in Table 1.

The design of wet wipes, the often long term storage of partly used packs, evaporation of the solution, and the interaction between the various components of the wipes and packaging makes them more susceptible to contamination than most other personal care products.

ABSTRACT

Wet wipes have become an increasingly popular item of everyday use in homes and workplaces. Although they were originally developed as personal care products, their success has led to the development of many products with household and industrial applications for cleaning, sanitising and polishing.

The preservation of wet wipes presents a number of challenges not encountered with other liquid products and often requires a unique solution incorporating a combination of different preservatives to

provide complete protection against microbial contamination.

The standard concern with matching a preservative to a particular formulation is further complicated by interactions between the liquid and substrate and the preservative and wipe substrate. A variety of different packaging types also further complicates the situation.

On top off these fundamental problems, the current trend in marketing personal care products that contain no controversial preservative ingredients is leading to further difficulties for the formulator. The requirement of products to contain no parabens, no formaldehyde donors and preferably no preservatives is becoming more widespread and mainstream.

Removing these long used and well understood actives from the formulators' range of acceptable ingredients is necessitating a completely new approach to the preservation process. The use of emollients and cationic actives to enhance the effect of traditional preservatives, or even replace them, is becoming more common.



Wet wipe manufacturing process

Wet wipes consist of two parts; a fabric carrier (wipe) impregnated with a solution (liquor) containing ingredients specific for the desired application. The wipe holds and spreads the liquid or collects and holds dirt or other matter that is being removed.

Fungal growth is often regarded as the main problem when preserving wet wipes

as when it occurs it is highly visible and wet wipes are more susceptible to fungal growth than typical personal care or household products. However, testing has shown that wet wipes are just as susceptible to growth of bacteria but as bacterial contamination is usually not visible its presence goes unnoticed (see Fig. 1).

Wet wipe substrates

The nature and composition of the wipe can have an effect on preservation/wipe interaction as well as subsequent preservative system performance. The fibres used to produce the wipe may be natural or synthetic, with natural fibres often providing more preservation difficulties (see Table 2).

Wet wipes may consist of traditional woven fabrics but the cost of these has restricted their use resulting in most wet

Table 1: Wet wipe uses.

Personal care	Household/industrial
- Baby cleaning - Adult cleaning - Make-up removal - Skin moisturising - Sunscreen application	- Household cleaning and polishing - Hard-surface disinfection - Antibacterial wipes - Pet care - Industrial cleaning and sanitising

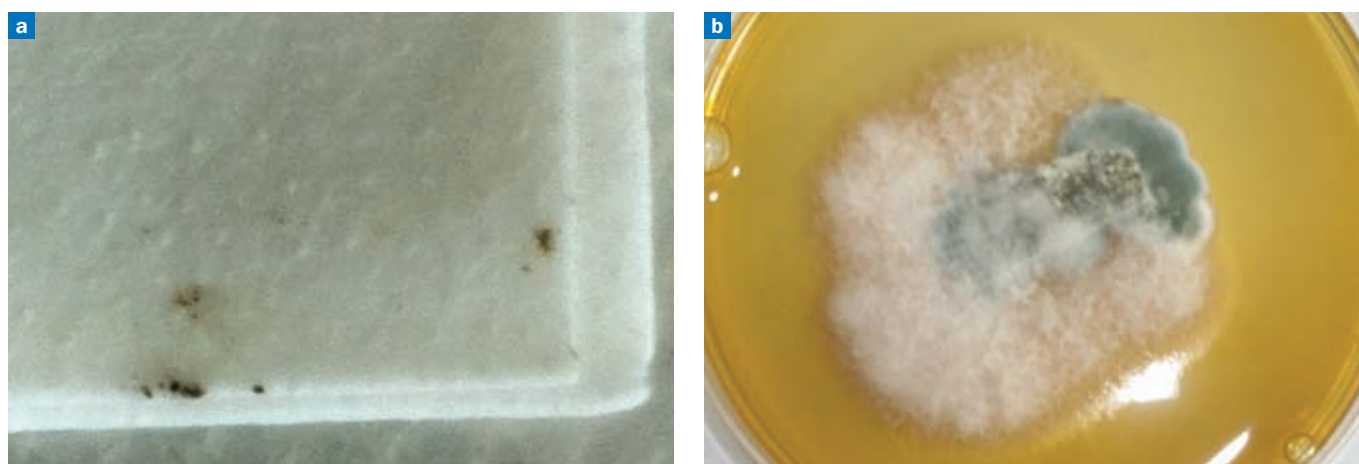


Figure 1: a) shows a wet wipe with small areas of fungal growth. b) shows the resultant growth when a small piece of the wet wipe was placed onto a malt extract agar and incubated.

wipes utilising non-woven materials. Non-woven fabrics are broadly defined as sheet or web structures bonded together by entangling fibre or filaments mechanically, thermally or chemically. They are engineered fabrics that are produced for specific applications that may include disposable for single-use, very durable fabric for long term use or anywhere in between.

The manufacturing process and raw materials used in the production of a non-woven fabric determines specific functions like liquid repellence or absorbancy, elasticity, softness or coarseness, strength and washability. Although natural substances such as cellulose may be used, most are now produced from synthetic fibres with polypropylene and polyesters (PET) as the major raw materials. Other materials including paper pulp, viscose (a chemically modified cellulose product), polyamides and cotton may be used to give the desired features to the final product.

There are a number of technologies for the production of non-woven fabrics, the major processes being airlaid and

spunlaced with some waterlaid products still produced.³⁻⁶

Airlaid

Air-formed fabric was first developed and commercialised in the early 1980s. Compared to normal paper, airlaid paper is claimed to be softer, bulkier and more porous while being stronger and having a textile-like surface and drape.

Unlike the traditional paper making process that uses water as a carrier for the fibres, airlaid involves the dispersion of the fibres in an air stream that is directed to deposit the fibres onto a moving belt. This forms a web that is then thermally bonded or spray bonded with resin, often acrylic polymer, and cured. Fibrous and powder material can be combined to produce single or multiple layer products, where each layer is specifically engineered for its application in the web, such as acquisition, distribution and absorption layers. Different fibre types may be overlaid to give a composite substrate with each layer having specific required properties.

Spunlaid

A variant of the airlaid process, spunlaid, produces non-wovens in one continuous process where fibres are spun and then directly dispersed into a web by deflectors or with air streams. This technique leads to faster belt speeds and cheaper costs.

Spunlace or spunjet

The spunlace or spunjet process (or hydroentanglement) is a variation of the spunlaid process and uses high speed jets of water to punch through a fibre web on a moving belt so the fibres become entangled. This entanglement of the fibres works as the bonding. The water is removed giving a substrate with soft handle, high strength and 'drapability'. Production of spunlace nonwovens is growing at a higher rate than other technologies.

Wetlaid

The third technology is wetlaid that resembles traditional paper making except using synthetic fibres. The process involves a slurry of water and fibres deposited onto a moving screen and then dewatered to form a web. The difference lies in the amount of synthetic fibres present in a wetlaid non-woven. The web is often impregnated with binders and consolidated by pressing between rollers.

The strength of the random oriented web is rather similar in all directions in the plane of the fabric. A wide range of natural, mineral, synthetic and man-made fibres of varying lengths can be used.

In all cases the resultant mat is then wound onto large rollers for the subsequent wet wipe production.

Formulation – liquid solution, the liquor

The liquor types used for personal care products fall broadly into three categories that are set out in Table 3, along with typical ingredients.

Table 2: Crucial steps in wet wipes production.

- Substrate used
- Formulation – liquid solution
- Preservatives
- Production process – GMP, plant hygiene, equipment
- Packaging – different types
- Testing – microbiological test method

Table 3: Formulations.

Type	Ingredients
Alcohol	10%-15% alcohol in water + surfactant
Water based	Water + surfactant + emollient + functional additives + perfume, chelating agents, pH buffers and preservative(s)
Emulsions	O/W or W/O emulsions (silicones, oils, waxes) + functional additives + perfume, chelating agents, pH buffers and preservative(s)

Table 4: Required preservative properties.

- Effective against a wide range of microorganisms
- Effective at liquor pH
- Stable in liquor pH
- Good compatibility with liquor formulation
- Not bonded or absorbed by fabric fibres
- Water soluble
- Heat stable if needed
- Good toxicological profile
- Globally approved and good acceptance by NGOs
- Cost effective

Table 5: Factors influencing performance of preservative.

- Formulation of the wet liquid
- Type of nonwoven
- Ratio of liquid:nonwoven carrier
- Distribution of wet liquid onto non-woven
- Preservative active
- Quality of raw materials
- Ionic bonding of preservative to the fibres
- Emulsion phase separation within the fibres
- Migration of liquor and preservatives within wipe stacks and fibres
- Effect of fibres on pH
- Fibre finishes leaching into the wipe liquor

While the first two categories are mainly for cleansing, the emulsion based products normally contain no surfactants and offer real skin care effects beyond cleaning.

The preservation of wet wipes

All aqueous and emulsion-based products require a preservative to protect them from bacterial and fungal contamination. Alcohol based wipes with an alcohol content in excess of 15% may be self preserving and generally require no additional preservation, but should be tested to be certain. Requirements for preservatives to be used in wet wipes are set out in Table 4.

The liquid phase of a wet wipe can be treated as any other personal care product when it comes to preservative selection and the usual parameters of preservative compatibility must be followed. However, it is vital to consider the wipe material as well when choosing a preservative. Some substrates that contain natural or some synthetic fibres can bind or absorb certain preservative actives reducing their efficiency. Because of this, it is important that both the solution and the formulated wet wipe are tested for preservative effectiveness.

As with all personal care products preservatives are added to protect the finished product from contamination by the end user and care must be taken to ensure that raw materials used in the production of the liquid are selected in the same manner they would be for any other type of personal care product. However, extra care must be taken to consider the source of the carrier material and the production processes. Any wipe material that has been produced by a wet process is liable to contain either bacterial or fungal spores. Incorrect storage of the bulk material may also make it liable to further contamination. Excessive levels of contamination will reduce the preservative content available to protect the finished wipe in use and will also be a source of

contamination of the finished packaged wipe (see Table 5).

Preservatives in use

The same familiar preservative actives used in other personal care products are used to protect wet wipes. However, there is an obvious tendency to use blends of many actives, and at high concentrations.

Most manufacturers use combinations or blends of preservatives with a variety of actives at high concentrations in an effort to provide good long term preservation. Commonly used combinations of preservatives are shown in Table 6.⁷

● Parabens

While the parabens are among the safest preservatives for personal care use their image has been severely tarnished by numerous NGO campaigns and their use is in decline for personal care products. However, they are still widely used in both personal care and household products. They are good fungicides, which make them very useful for wet wipes preservation, but are less effective against bacteria requiring their use in combination with other preservatives. They also have water solubility concerns that can make them difficult to use or require they be dissolved in a solvent such as phenoxyethanol for easier addition.

● DMDMH

DMDMH is one of a group of formaldehyde donors so it is also under public pressure to be removed. As with all formaldehyde

donors there is limited activity against fungi but good efficacy against bacteria.

● Bronopol

Bronopol is an effective broad spectrum preservative with better activity against bacteria than fungi. It is often claimed to be a formaldehyde donor but does not express its antimicrobial activity by formaldehyde release. It may cause discolouration of the finished product and does face regulatory pressure due to the possibility of nitrosamine formation in the presence of TEA.

● Isothiazolinones

The isothiazolinones are a diverse group of preservatives with great variation in stability, water solubility and preservative efficacy:

Benzisothiazolinone: The first to be developed was benzisothiazolinone (BIT) and it has been in use in household and industrial applications for over 40 years. It has been proposed as a personal care preservative but its approval and addition to Annex V of the EU Cosmetics Regulation is still pending. BIT is effective against most bacteria but has an activity gap against *Pseudomonas* sp. and is poor against fungi. This can be overcome by using higher concentrations or in combination with methylisothiazolinone.

Methylchloroisothiazolinone (MCI)/Methylisothiazolinone (MI): MCI/MI has been widely used in personal care products but has a potential to cause skin sensitisation, primarily due to the MCI, so is limited by the EU Cosmetics Regulations

Table 6: Preservative used for wet wipes.

Parabens	DMDMH	Bronopol
Iodopropynylbutylcarbamate (IPBC)	Organic acids (Sodium benzoate and potassium sorbate)	
Phenoxyethanol	Benzyl alcohol	Glycols
Methylchloroisothiazolinone + methylisothiazolinone	Polyaminopropylbiguanide (PHMB)	Benzisothiazolinone
Methylisothiazolinone	Quaternary ammonium compounds	

and ASEAN Cosmetic Directive to 15 ppm for leave-on and rinse-off applications. It is widely used for shower and hair products and offers complete protection against both bacteria and fungi. Although it is permitted, this combination is not often used in leave-on products.

Methylisothiazolinone: This is one of the most recent preservatives to be added to Annex V of the EU Cosmetic Directive and has also been included in the updated Annex VI of the ASEAN Cosmetics Directive. MI is very effective against bacteria but weak against fungi requiring it to be always used in conjunction with other preservatives. It has been very successfully combined with BIT and overcomes the poor fungal efficacy while also showing excellent activity against *Pseudomonas* sp. The combination cannot be used for personal care wipes for supply in countries with regulations based on the EU Cosmetic Regulations or the ASEAN Cosmetics Directive, but may be used in household and industrial applications.

MI has also been combined with parabens, PHMB, IPBC and chlorophenesin to overcome its poor fungal efficacy. Combinations with decylene glycol have also shown excellent preservative ability. Testing has shown that methylisothiazolinone is probably the most effective preservative for wet wipes when used as part of a preservative combination.

● IPBC

IPBC is a true fungicide with negligible efficacy against bacteria. It is used in combination with other preservatives that lack good fungal activity or where the finished product is particularly prone to fungal attack. The EU Cosmetics Regulation was altered to restrict the use of IPBC in preparations for use on children under three years-of-age (except in bath products/shampoos and gels) and in products used over large areas of the body and these changes have been adopted in the updated Annex VI of the ASEAN Cosmetics Directive. This was due to concerns of iodine intake that the SCCP concluded should not exceed 20% of the recommended daily intake of 150 µg.^{8,9} Like many parts of the world the populations of ASEAN countries are deficient in iodine and add it to food stuffs such as salt to increase the daily intake.¹⁰

This loss of an efficient fungicide is particularly worrying as the conditions within the packaged wet wipes tend to favour the development of moulds and yeasts, particularly where 'skin compatible' pHs of 5-6 are used. The parabens are very effective against fungi but with their decreased use of specific fungicides may

Table 7: Preservative blends used for wet wipes

Phenoxyethanol + parabens + bronopol	MCI/MI + parabens
Phenoxyethanol + DMDMH + IPBC	MI + parabens
Phenoxyethanol + IPBC	DMDMH + parabens
MIT + chlorophenesin	Benzyl alcohol + PHMB + MCI/MI
Phenoxyethanol + sodium benzoate + dehydroacetic acid	Dehydroacetic acid + benzoic acid + potassium sorbate + benzyl alcohol
Phenoxyethanol + sodium benzoate + dehydroacetic acid + benzoic acid	Phenoxyethanol + sodium benzoate + benzyl alcohol

Table 8: Loss of PHMB on viscose wipes.

Sample	PHMB content (ppm)			
	Time (days)	0	4	8
Control (water + 3000 ppm PHMB)		3,000	2,850	3,144
Viscose + 3000ppm PHMB		3,000	1,264	1,445
Polypropylene + 3000ppm PHMB		3,000	2,726	3,242

be required to help control the fungi and the removal of IPBC limits available solutions. Chlorophenesin is a viable alternative but can be difficult to use due to low water solubility.

IPBC can be still be used in personal care wet wipes in Australia where regulations do not prohibit its use, and for non personal care wet wipe products in all countries.

● Phenoxyethanol

The data presented in Table 7 indicates that the use of phenoxyethanol is quite common, possibly due to its useful properties and lack of controversy. It has good activity against *Pseudomonas* sp. while acting as a solvent for other actives. It lacks efficacy against fungi and needs to be used at high concentrations if used alone. Phenoxyethanol is widely used in combination with the parabens, formaldehyde donors, bronopol, IPBC and MCI/MI, usually with at least two other actives.



● PHMB

There are problems with the use of some preservative actives. Apart from the usual issues of dissolving in water and migration to the oil phase in emulsions there is also the possibility of absorption onto the carrier or container. Polyaminopropylbiguanide (PHMB) is gaining popularity, particularly for antibacterial wipes, but due to its strongly cationic nature there is the risk of being absorbed onto anionic viscose or cellulose fibres, so it must not be used with viscose or viscose blend wipes. Table 8 shows results of work conducted where 10 gm of fabric was soaked in 100 mL of PHMB solution (3000 ppm) and the liquid analysed for PHMB content after four and eight days' contact. The results clearly show a significant loss of PHMB when it is exposed to viscose.¹¹

Antibacterial wipes will often contain PHMB or other cationic substances such as quaternary ammonium salts as the antimicrobial active ingredient that will also double as the preservative. Care must be taken when using these to ensure they are not bound to the wipe and thus unavailable to act against microorganisms.

These products usually make simple claims such as 'antibacterial, kills 99.9% of germs'. These products may be regulated in some countries, depending on specific claims made on labels.

● Organic acids, alcohols and glycols

There has been an increase in the use of organic acids and alcohols, with glycols and emollients being used as biocide boosters. The use of organic acids requires an acidic pH for the liquid phase to ensure the acids remain active. Unfortunately this pH increases the susceptibility of the wet wipes to fungal contamination, as they prefer an acidic environment. Also, some

wipes affect the pH of the liquid raising it by half to one pH unit. This is particularly important when using airlaid wipes where interaction between the coating polymer and liquor may raise the pH. If organic acids are being used this may be sufficient to raise the pH above the pK_a value of the acid and reduce the efficacy of the preservatives. Therefore the pH needs to be controlled in the finished product, not just the liquid, and should be checked in liquor expelled from the wipe. Although the organic acids may be used on their own in combination, they are very often used in combination with phenoxyethanol.

Glycols, in particular the 1,2 alkane glycols, are becoming more widely used in personal care products and their use in wet wipes is also increasing. The glycols lower water activity and some offer specific antimicrobial properties by interfering with the cell membrane of microorganisms. The two most common used are caprylyl glycol and decylene glycol. They are not classified as preservatives and are listed as humectants or emollients, imparting sensory characteristics to the skin. They offer relatively good antimicrobial properties against both bacteria and fungi but only have moderate activity against microorganisms when used on their own. They may exhibit compatibility issues in O/W emulsions, have low water solubility and can be expensive.

The EU Cosmetics Directive Annex VI contained a preamble as follows:

1. *Preservatives are substances which may be added to cosmetic products for the primary purpose of inhibiting the development of microorganisms in such products.*

3. *Other substances used in the formulation of cosmetic products may also have antimicrobial properties and thus help in the preservation of the products, as, for instance, many essential oils and some alcohols. These substances are not included in the ANNEX.*

The recast EU Cosmetics Regulation updated the preamble to read:

Preservatives mean substances which are exclusively or mainly intended to inhibit the development of microorganisms in the cosmetic product.

This was changed to control the practice of using non-listed preservatives under the guise of including them for another purpose. Unfortunately the revised Annex VI of the ASEAN Cosmetics Directive has not included this change. As the glycols are not approved as preservatives, care needs to be exercised in using them in personal care wet wipes that will be exported to countries that follow the EU Regulation.



Figure 2: Wet wipe packaging styles.

While there is a move away from traditional preservatives such as the parabens, formaldehyde donors, IPBC and MCI/MI in personal care products, they are still used often in wet wipes. However, there is a push to move away from these preservatives particularly for natural products, although this market appears to be lagging behind other sectors of the personal care market. The traditional preservatives are still widely used in household wet wipes.

There is no universal preservative system for use in wet wipes. Wet wipes are difficult to preserve and care must be taken to ensure a robust system is chosen.

Production process

Large diameter rolls of wipe substrate are fed into machines where they are slit to the required width. The non woven cloth is fed into a coating machine where the liquid phase is applied by a number of methods: running the non-woven through a trough of the solution, spraying sheets with the liquid through a series of nozzles or injecting into packs of folded wipes. Addition of the liquid to the wipes in a manner that gives the most even distribution of the liquid to the wipes provides the best protection to the finished product.

Injection into a folded pack gives the least uniform distribution while spraying liquor from above onto the moving wipes gives the most uniform distribution. The amount of liquor on a wipe is also critical for its effectiveness and the ratio between liquor and wipes varies for different applications. For cleaning purposes either too little or too much liquor is detrimental.¹²

Packaging

Wet wipes may be packaged in a number of ways, depending on the eventual use of the wipes. The preferred method from a preserving point of view is individual sachets. Once packed there can be no introduction of microorganisms and no loss of moisture. Unfortunately this is the most

expensive and inconvenient way to supply wet wipes and so they are generally supplied in bulk packs. Household wipes are usually packed into soft packs or tubes with pull through lids while baby wipes are most often in tubs with an opening lid for easy access. Facial cleansers are often in soft packs with a small resealable opening. Figure 2 shows a variety of typical pack types.

The different packaging effects the migration of the liquid on the wet wipe but all will allow migration away from the opening. For rolled wipes in tubes, the tops of all wipes may experience drying and increased chance of microbial growth, while with tubs and soft packs the wipe nearest the opening will be most susceptible. Tubs with a large opening lid that may be left open allow greater chance of ingress of dirty fingers and microbes from the air than do the pull through tubes so may require more robust preservation. They are also most likely to be able to be refilled with new wipes so any microbial contamination from the previous load may get a head start on growing on the refills.

Figure 3a shows a wet wipe protruding from a canister that appears normal. However opening the canister (Fig. 3b) reveals light fungal growth on the top of the wet wipes, but when the roll is removed from the canister (Fig. 3c) extensive fungal growth is evident. The exposed wet wipe in Figure 3a was quite dry and unable to support fungal growth, while the wipes inside the canister maintained sufficient moisture for fungi to grow. When removed from the canister the top of the wipes roll was drier than the base as the liquor had migrated towards the bottom of the canister. This migration resulted in the wipes near the top of the canister containing sufficient moisture for fungal growth but insufficient preservative to control them while the wipes towards the bottom of the container had sufficient preservative to stop the fungi from growing.

Preservative efficacy testing of wet wipes

There are a range of test methods currently in use, all with the basic concept of adding microorganisms and checking for survival. Early methods only tested the liquor and it has been shown that this was not appropriate.¹³ As already discussed, interaction of the liquid with the wipes may affect the efficacy of the preservative system, or heavily contaminated wipe feedstock could reduce the preservative concentration resulting in field failures in products where the liquor had passed antimicrobial preservative efficacy tests. It has also been found that preservatives may interact with the packaging reducing their



Figure 3.

effectiveness. Newer test methods rely on adding a large number of different organisms to the finished wipes and storing them under appropriate conditions for the desired length of time. Surviving organisms are then enumerated by collecting the liquor from the wipes and conducting microbial counts using various techniques.

The manner of inoculation varies with one published method requiring collecting the test organisms on membranes, drying them and placing the dried membranes between two wipes in the original pack.¹³ Typically the inoculum is sprayed or pipetted onto the wipes. It may consist of pure cultures, with each test organism introduced to a separate test piece, or a mixture of specified test organisms added together. The manner in which the organisms are added to the test piece and treatment to ensure even distribution of the organisms has a huge effect on the test outcome and the reproducibility of the test results.

The concentration and type of organisms is also critical; too low a number may give a false sense of good preservation, while an excessive concentration may require unnecessarily high levels of preservative to pass the test. The use of inappropriate test strains or strains with known preservative tolerance may also give misleading results.

The test method must be designed to place the inoculum in an appropriate part of the wipes and ensure good recovery of surviving organisms. The recovery method is as critical as the inoculation to ensure that results achieved are truly representative of the number of organisms surviving on the test pieces. It is also necessary to develop pass criteria based on the reduction in the number of organisms surviving after specified time periods.

The selection of an appropriate test method is critical to ensure confidence that a test result from the laboratory will truly represent what will happen in the field when the product is produced and released.

Regulatory aspects

Wet wipe preservatives, depending on their application and claims, are required to meet different regulatory requirements. Wet wipes may be used for purposes with Therapeutic claims, such as sunscreens, or other applications, like disinfectants, that may bring them under regulatory control depending on which country they are to be sold in. Preservatives used in personal care wipes will need to meet the requirements of the ASEAN Cosmetics Directive or the EU Cosmetics Regulations if they are to be exported to an EU country.

Preservatives used in industrial and household applications are not limited by these restrictions. Thus the range of preservative actives available for a surface cleaning wipe can be different from a skin cleaning wipe even though the exposure risk may be similar to the person using the wipes.

The influence of Eco-labels can further complicate the situation through more stringent criteria limiting preservative use. This makes it impossible to make global recommendations for preservatives in consumer applications where wet wipes predominate.

Conclusion

The preservation of wet wipes is a complex problem due to influences of the wipe and packaging on the liquid phase of the wet wipe. Generally high concentrations of a number of preservative actives are required to ensure complete long term protection against bacterial and fungal contamination.

For industrial and household applications simple systems using traditional preservatives such as MCI/MI, MI/BIT and blends with formaldehyde donors are still widely used.

The use of methylisothiazolinone in combination with other preservatives has been found to offer excellent protection to most personal care wipes and the use of phenoxyethanol is quite widespread.

A move towards 'natural' products is occurring at a slower pace than other personal care sectors and the use of

phenoxyethanol in combination with the organic acids may provide good protection as long as the correct pH is maintained. The addition of multifunctional ingredients, such as emollients and humectants which act as biocide boosters, improves the preservation of wet wipes ensuring good long term stability.

Proven preservatives such as parabens, IPBC and methylchloroisothiazolinone/methylisothiazolinone are still being used but with limitations.

● A version of this paper was presented at the Australian Cosmetic Chemists Conference in Adelaide, 2012.

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