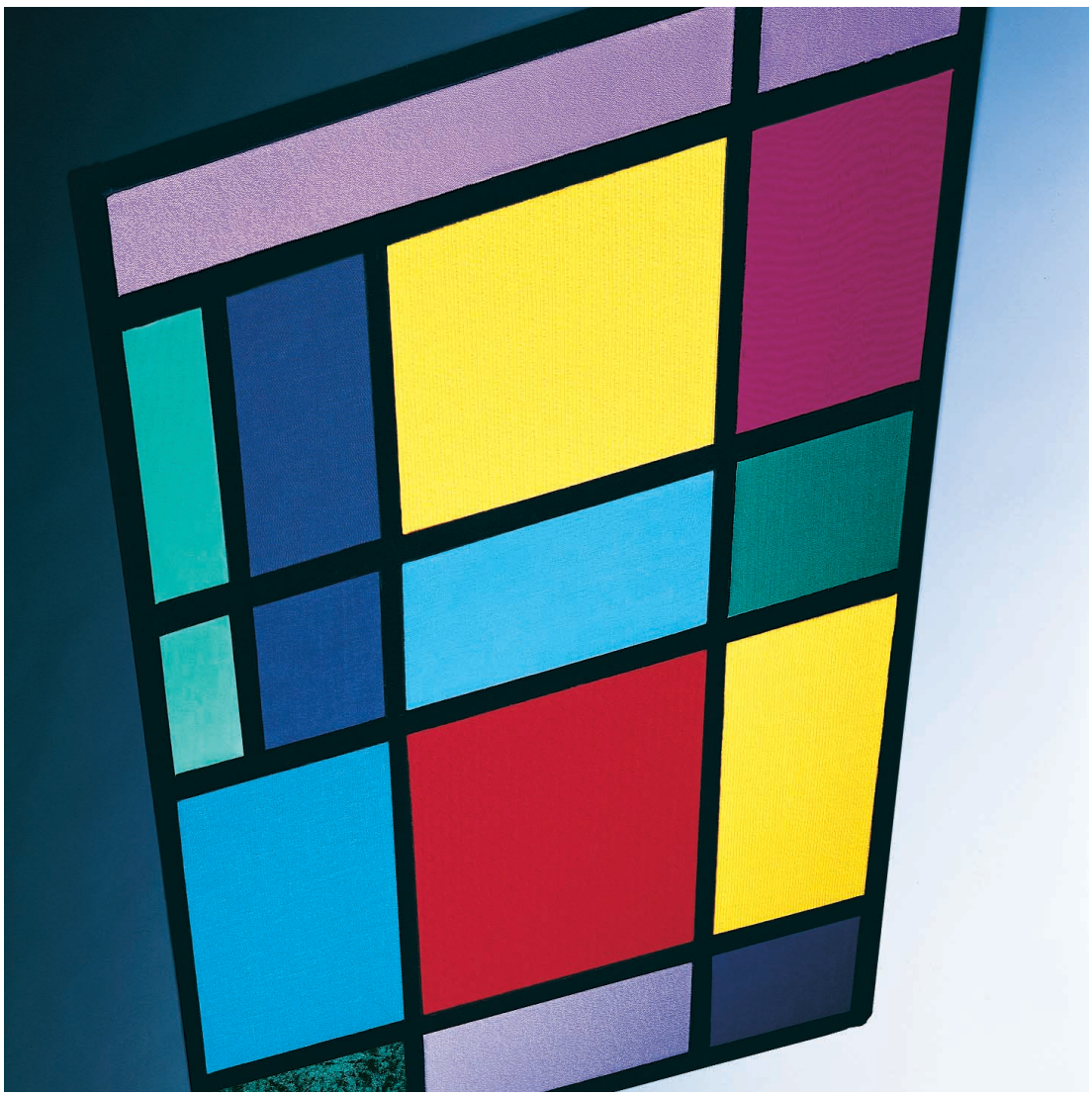
Technical Information

TI/T 344 e
January 2000 (RV)

BASF products for resin finishing

brilliant SOLUTIONS...
WITH TEXTILE CHEMICALS
FROM BASF



Colorants and Finishing Products

BASF

1 General information on finishing

Finishing is generally the last stage of modern textile production. Its goal is to convert the bleached, dyed or printed fabric by mechanical and chemical treatment into a suitable state for sale and making-up. There are three types of finishing process, mechanical, non-permanent and washfast, each of which usually involves the continuous treatment of woven or knitted fabrics in open-width form. This leaflet focuses on the washfast finishing of woven and knitted fabrics composed of cotton, other cellulosic fibres, and their blends with synthetic fibres. The most important aspect is chemical finishing, also known as resin finishing, easycare finishing or wash-and-wear finishing. Resin finishing has been able to maintain its position in the finishing of textiles based on cellulosic fibres despite various disadvantages such as strength losses, shade changes, reduced whiteness, and controversy about formaldehyde content. In fact, recently there has been a resurgence in its importance, because it allows textile finishers to stand out from competitors by producing fabrics with enhanced quality.

The advantages of resin-finished over unfinished textiles, especially after washing, are:

- **improved dimensional stability and shape retention**
- **less tendency to creasing**
- **easier to iron**
- **softer and smoother**
- **better appearance and therefore more durable**
- **less change in shade**
- **improved wet fastness of dyeings and prints**
- **less tendency to pilling, especially of fibre blends**
- **greater wash resistance of mechanically produced lustre and embossed finishes and finishes with softeners, stiffening agents, water-repellents and oil-repellents**

The recipe for a resin-finishing liquor normally consists of:

- **Crosslinking agent**
- **Catalyst**
- **Additives**
- **Surfactants**

Crosslinking agents

Resin finishing is carried out with products known as crosslinking agents. These change woven and knitted fabrics composed of cellulosic fibres and their blends with synthetic fibres in such a way that the resulting textiles are easier to care for.

Catalysts

Another essential component of the resin-finishing recipe is the catalyst. It allows the reaction to be carried out within the 130–180 °C temperature range usually employed in the textile industry, and within the usual curing times (several minutes in the case of curing machines and several seconds in the case of stenters).

Additives

The purpose of the additives is to offset partly or completely the adverse effects of the crosslinking agent. Thus softening and smoothing agents are applied not only to improve the handle, but also to compensate as much as possible for losses in tear strength and abrasion resistance. Other additives serve to impart a particular character to the fabric. Examples are stiffening and filling agents, water-repellents, hydrophilizing agents, etc.

Surfactants

Every resin-finishing recipe contains surfactants as emulsifiers, wetting agents and stabilizers. These surface-active substances are necessary to ensure that the fabric is wet rapidly and thoroughly during padding and to stabilize the recipe components and liquors.

2 General information on crosslinking agents

The first resin-finishing agents – developed in the 1930s to improve the poor wet strength of viscose staple fabrics – were compounds of formaldehyde and urea, though compounds of formaldehyde and melamine were later also used. In recent years the importance of this group of crosslinking agents has declined because of the controversy about the high levels of free formaldehyde in the products and on the finished goods.

With the appearance of easycare synthetic fibres in the 1950s, heterocyclic crosslinking agents were developed to improve the competitiveness of cotton. Heterocyclic crosslinking agents are based on urea, formaldehyde, and various other substances such as diamines and, in particular, glyoxal.

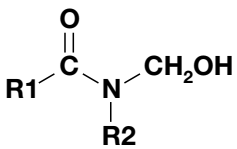
Of the crosslinking agents that are formaldehyde-free, only products made from dimethyl urea and glyoxal have gained a small share of the market. Other formaldehyde-free crosslinkers have remained insignificant because of high toxicity, high manufacturing costs, inadequate wash resistance or poor effects.

Since practically all effective resin-finishing agents are based on formaldehyde, they have become part of the public debate about the toxicity of this substance. This debate has strongly influenced and stimulated the development of crosslinking agents in recent years. New crosslinkers for low-formaldehyde finishing and formaldehyde-free resins were developed.

With the revival of resin finishing, processes that were almost forgotten, such as postcuring and moist crosslinking, are being applied again.

2.1 Crosslinking agents

Crosslinking agents for formaldehyde-based resin finishing have the following general formula:



They are divided into so-called “self-crosslinking” and “reactant crosslinking” agents. Self-crosslinking agents have a reactive hydrogen atom on the nitrogen atom (R2 = H), whilst in reactant crosslinking agents the nitrogen is part of a heterocyclic ring and therefore does not carry a reactive hydrogen. Crosslinkers based on melamine represent a transition between self-crosslinking and reactant crosslinking agents. In BASF’s range of finishing agents, the self-crosslinking agents are called **Kaurit**® types and the reactant crosslinking agents **Fixapret**® types.

Kaurit types

The Kaurit types are reaction products of urea and formaldehyde or melamine and formaldehyde. Some of these compounds are also methylated with methanol. They are used mainly for resin finishing textiles of regenerated-cellulose fibres, and more generally for applying a stiffening finish. Particularly suitable for this purpose are the N-methylol compounds of urea, which impart a good swelling resistance to the fabric. They also improve the dimensional stability and crease recovery of regenerated-cellulose fabrics, giving them a high level of resilience. Chemical modification

of these compounds and adaptation of the reaction conditions have allowed similar effects to be obtained on cotton and its blends.

Fixapret types

The Fixapret types are heterocyclic reaction products of urea, glyoxal and formaldehyde. Some of them are also modified with alcohols. Fixapret NF, which is **formaldehyde-free**, is exceptional in that it is a compound of dimethyl urea and glyoxal. Among the formaldehyde-free crosslinking agents, only those that are compounds of this type have achieved limited success in the resin-finishing market. All the Fixapret types are suitable for resin finishing woven and knitted fabrics composed of cellulosic fibres and their blends with synthetic fibres.

The Fixapret types are reactant crosslinkers. Since, unlike self-crosslinking agents, they have no reactive hydrogen atom on the nitrogen atom, they do not tend to condense in the finishing liquor if this is left to stand for an extended period, even in the presence of a catalyst. It is mainly for this reason that they produce a much softer fabric handle than self-crosslinking agents. The Fixapret types are very stable to hydrolysis both in the liquor and on the fabric, which means the liquors conform to the maximum workplace concentration limits for formaldehyde even on long standing, and the easycare effects are resistant to repeated washing and dry cleaning, and to storage under tropical conditions.

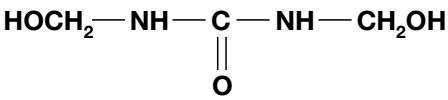
The Fixapret range has been thoroughly overhauled over the last few years. The new generation of Fixapret types produce finishes with an extremely low level of free formaldehyde on the fabric. These crosslinking agents are nevertheless very reactive even with magnesium chloride as the catalyst, making higher production speeds and lower production costs possible.

The Fixapret types without exception contain only a very small amount of chemically uncombined formaldehyde, so that if applied properly they allow the legal limits on formaldehyde at the workplace and in the exhaust air to be met. Those designed for low-formaldehyde finishing produce finishes with a low to very low level of free formaldehyde on the goods. Naturally, formaldehyde-free Fixapret NF causes no problems with formaldehyde at all.

BASF offers the following crosslinking agents:

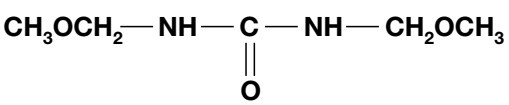
- **Kaurit S**
- **Kaurit W**
- **Kaurit M 70**
- **Fixapret AP**
- **Fixapret CP Conc.**
- **Fixapret CNR Conc.**
- **Fixapret CV**
- **Fixapret CL**
- **Fixapret CM**
- **Fixapret ECO**
- **Fixapret NF**

Kaurit S



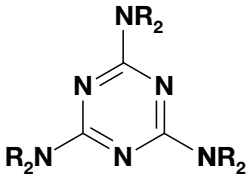
Kaurit S is a white, crystalline powder of dimethylol urea. It finds application where maximum resilience is required, e.g. on all types of pile fabrics (cord and velvet), regenerated-cellulose fabrics, and polyester/regenerated-cellulose blends. The product's poor stability to hydrolysis leads to high levels of free formaldehyde. Kaurit S is very reactive, allowing curing at relatively low temperatures but giving it limited bath stability. Ammonium and metal salts serve as catalysts. Because of the high formaldehyde levels in the finishing bath and on the goods, this product is no longer as important as it used to be.

Kaurit W



Kaurit W is a concentrated aqueous paste of bis(methoxymethyl) urea. It has similar properties to Kaurit S but has better bath stability, is not so resilient and produces a somewhat softer handle. Kaurit W is ideal for giving a stiffening finish to polyamide ("petticoat effect"). Polyamide textiles finished with Kaurit W give low formaldehyde values in the AATCC 112 and LAW 112 tests (EN ISO 14184 Parts 1 and 2).

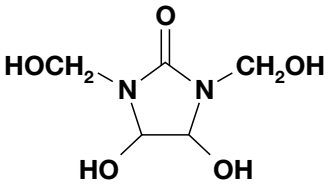
Kaurit M 70



R = H, CH_2OH, CH_2OCH_3

Compared with reactant crosslinkers, Kaurit M 70 has a good buffer effect and thereby minimizes strength losses in sensitive qualities. It is therefore often applied in small quantities with other crosslinkers. Kaurit M 70 is particularly effective in improving the permanence of water-repellent, oil-repellent and calender finishes. Furthermore, it is the only filling and stiffening agent that produces wash-resistant effects on textiles containing polyester. Thus it is very suitable for stiffening industrial textiles. But like the other Kaurit types, it has lost importance because of the high levels of free formaldehyde in the product and on the fabric.

Fixapret CP Conc.



Fixapret CP Conc. is a concentrated aqueous solution of a reaction product of urea, glyoxal and formaldehyde. It is usually known as 1,3-dimethylol-4,5-dihydroxyethylene urea (or DMDHEU), although the systematic chemical name is 1,3-bis(hydroxymethyl)-4,5-dihydroxyimidazolidinone-2. With the exception of Fixapret NF, all Fixapret types are based on this compound. DMDHEU-based crosslinkers are now by far the most significant group in the industrial countries, occupying approx. 80 to 90 % of the crosslinker market. In the 1950s, BASF contributed greatly to the development and large-scale production of this crosslinker group. DMDHEU is highly reactive and very suitable for wash-resistant resin finishing. However, in many cases the free-formaldehyde levels produced by this crosslinker on the fabric no longer meet today's high standards.

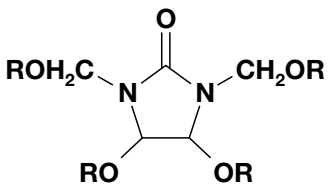
Fixapret CNR Conc.

Fixapret CNR Conc. is a concentrated standard crosslinking agent similar to Fixapret CP Conc. but containing diethylene glycol (DEG) as an additional component. The finishes give low formaldehyde values in the Shirley I test (also known as the "Free Formaldehyde" test or BS 6806 Part 2). Fixapret CNR Conc. is very suitable for the resin finishing of regenerated cellulose.

Fixapret CV

Fixapret CV, a concentrated aqueous solution, is a further development of Fixapret CNR Conc. It is a top-quality product for minimum formaldehyde values in the Shirley I test ("Free Formaldehyde" test or BS 6806 Part 2) and ideal for the resin finishing of regenerated cellulose and its blends. Because of the product's high reactivity, catalysis with magnesium chloride is sufficient on cotton, and often also on viscose.

Fixapret CL



R = H or CH_3

Fixapret CL is the concentrated aqueous solution of a reaction product of DMDHEU with methanol. Compared with earlier crosslinkers of this type, it yields particularly low formaldehyde values in the LAW 112 and AATCC 112 tests (EN ISO 14184 Parts 1 and 2) and a higher chlorine resistance. The reactivity has also been improved to such an extent that Fixapret CL can be cured with magnesium chloride as the catalyst under the same conditions as normal DMDHEU. It is ideal for the moist crosslinking process.

Fixapret CM

Fixapret CM is similar to Fixapret CL, but also contains DEG. The chlorine resistance, formaldehyde values and degree of whiteness obtained are better than with Fixapret CL.

Fixapret ECO

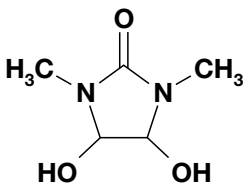
Fixapret ECO is a modified crosslinker based on DMDHEU, methanol and DEG. Since it contains extremely low levels of uncombined formaldehyde (less than 0.1%) and methanol, even relatively large amounts of Fixapret ECO in the finishing recipe permit the lowest emission limits to be observed. The formaldehyde levels on the fabric as measured by the three usual test methods are also extremely low, so that even the finishing of regenerated cellulose, which requires a large amount of crosslinking agent, produces formaldehyde values that fall below very low limits, e.g. 75 ppm in the LAW 112 test of Öko-Tex Standard 100.

Fixapret AP

The latest BASF development in the field of crosslinkers is Fixapret AP, which contains extremely little uncombined formaldehyde (less than 0.1%) and methanol. It produces the same low formaldehyde values on the fabric as Fixapret ECO and makes it even easier to observe the emission limits.

Fixapret AP is an essential component of the new ***A*dvanced *P*erformance finish** (AP finish). The corresponding process, for which a patent application has been submitted, produces a particularly high standard of finishing effects for an acceptable loss in fibre strength. It can be used to replace the moist crosslinking process, which until now has been the usual process for obtaining such effects but is difficult to carry out.

Fixapret NF



Fixapret NF is an aqueous solution of 1,3-dimethyl-4,5-dihydroxyethylene urea (DMeDHEU, systematic chemical name 1,3-dimethyl-4,5-dihydroxyimidazolidinone-2). This crosslinking agent is **free** of formaldehyde. It produces wash-resistant finishes with outstanding resistance to chlorine and a much softer fabric handle than that obtained with formaldehyde-based crosslinkers. Resin finishing with Fixapret NF and Condensol® N greatly reduces the usual strength losses. In the absence of other sources of formaldehyde such as fixing agents, preservatives and other formaldehyde-based products, the finishes obtained are formaldehyde-free. The use of Condensol N as the catalyst is strongly advised, because magnesium chloride tends to produce an odour and have an adverse effect on the degree of whiteness.

2.2 Crosslinking processes

The resin-finishing process involves padding and drying the open-width, cellulosic-fibre-based fabric and then curing it by one of a number of methods.

The following different crosslinking processes are used in resin finishing:

- **Dry crosslinking process**
- **Moist crosslinking process**
- **Wet crosslinking process**
- **Postcure process**
- **Precure process**
- **Dip-dry process**

Dry crosslinking process

The most important of these processes is dry crosslinking, in which the fabric is cured in a dry state. After being padded, the fabric is usually dried on the stenter and then cured in a curing apparatus, or on the stenter immediately after drying (flash-curing process).

Moist crosslinking process

In moist crosslinking, the fabric is cured in a moist, partially swollen state (about 6 –12 % residual moisture). The fabric is padded with a liquor containing a mineral-acid catalyst in addition to the crosslinker. BASF recommends sulfuric acid, because hydrochloric acid is problematic for various reasons including hydrogen chloride in the exhaust air. The fabric is subsequently dried to a residual moisture content of 6 –12 %. After being batched for one or two days at a temperature of 25 – 35 °C, the fabric is washed, neutralized and dried. Afterwards it is usually further treated with handle-finishing agents. The process is very expensive, because several drying steps are necessary. Moreover, in practice it is difficult to maintain the exact conditions required, such as residual moisture content and batching temperature. Very good easycare effects, low formaldehyde values and low strength losses are obtained only if these conditions are exactly maintained. The process has regained importance in recent years, following an interval of more than 20 years, in the manufacture of virtually non-iron, all-cotton textiles of acceptable strength.

Wet crosslinking process

In wet crosslinking, the reaction takes place when the fabric is in a wet, fully swollen state. Today this method is no longer used, even though it is much easier to carry out than moist crosslinking, because the dry crease recovery is almost the same as that of untreated cotton textiles. As in the moist crosslinking process, a liquor containing a strong mineral acid is applied and the batch of wet fabric is rotated for about 20 hours at room temperature. The fabric is then washed, dried, neutralized and, if necessary, aftersoftened. Because of the repeated drying required, this process is also expensive.

Postcure process

The postcure process is another old process that has experienced a revival, beginning in the USA. It belongs to the dry crosslinking methods and is the most significant permanent-press method. The fabric is treated as in standard dry crosslinking but not cured. The treated fabric is subsequently made up into garments and provided with crease lines or pleats in the steam press before being oven-cured. Earlier permanent-press methods, for example the Koratron process, have disappeared as a result of a number of disadvantages, such as a formaldehyde odour problem during steaming (more than 1000 ppm in the AATCC 112 test!) and a pronounced shade change because of the zinc nitrate that had to be used to achieve the necessary high reactivity. Today's postcure process can be carried out with low-formaldehyde crosslinkers that are reactive enough to be catalysed with magnesium chloride, and formaldehyde-free crosslinkers that have the additional advantage of producing an even better tensile strength (cf. Fig. 5).

Precure process

Crosslinking in the precure process is also carried out in the dry state. Another permanent-press method, it is a special case in which blended wovens of synthetic and cellulosic fibres (usually PES/CO or PES/CV with over 60 % PES) are provided with permanent creases. In the first step, the cellulosic component undergoes standard continuous resin-finishing by the dry crosslinking process. After making-up, the finished garment is shaped by heat setting the synthetic fibres at high temperature and under high pressure in special ironing presses.

Today, the name "precure process" is occasionally given to the dry crosslinking process to distinguish it from the postcure process. The original precure process is no longer important.

Dip-dry process

The dip-dry process is a special case of the permanent-press or postcure process. The fabric is first made up into garments, which are dipped to impregnate them with the finishing liquor, centrifuged, dried, ironed and cured. This process has also experienced a revival. However, the curing step is difficult because of the seams, problems with yellowing, etc.

2.3 Formaldehyde in resin finishing

Almost all crosslinking agents commonly used today contain formaldehyde as part of the molecule. Because of the on-going debate about its suspected carcinogenicity, formaldehyde takes a central position in ecological assessments of resin finishing agents, especially in the eyes of the public. This debate and the ensuing legislation have influenced and stimulated development work in this field, as formaldehyde-free crosslinkers, or ones that are as low in formaldehyde as possible, have been sought.

More than 20 epidemiological studies involving over 100,000 workers, some of whom had had 30 to 40 years of occupational exposure to high concentrations of formaldehyde, did not reveal any increase in the incidence of nasal or respiratory cancer. In July 1995, the Chemical Industry Institute of Toxicology (USA) published a further epidemiological study, involving foundry workers exposed to high levels of formaldehyde in the workplace. Again, an increased cancer risk was not discovered. Humans and animals are presumably habituated to low concentrations of formaldehyde because of its ubiquity. The substance is formed from hydrocarbons in the atmosphere by photochemical processes and is released by every combustion process. It is found as a metabolic product in food and even in blood, where it is used in the synthesis of essential substances such as hydroxyproline and hydroxylysine.

Most test methods determine not only actual “free” formaldehyde, but also chemically combined formaldehyde, which it is the purpose of the test method to liberate. There are four main test methods, of which three have gained general acceptance (Fig. 1). Only the DIN method determines merely the chemically uncombined formaldehyde. The other methods also determine the formaldehyde released by hydrolysis of N-methylol groups in the crosslinking agent, and the Shirley I test (BS 6806 Part 2 or “Free Formaldehyde” test) determines in addition that released by hydrolysis of N-methoxymethyl groups. The intention is to simulate unfavourable conditions when resin-finished fabrics are stored or being made up into garments (AATCC 112 or BS 6806 Part 3, which is a slightly modified version of AATCC 112), or the conditions created on the human skin when the garments are worn (Law 112 and Shirley I). The Shirley I test determines mainly formaldehyde derived from extracted, unreacted, intact crosslinker molecules. Since the extract is boiled in concentrated sulfuric acid in the presence of chromotropic acid, formaldehyde from N-methoxymethyl groups is obtained as well. Thus formaldehyde is formed that would never arise when resin-finished textiles are stored or worn. The usual designation, “Free Formaldehyde” test, is therefore a very misleading one. However, the Shirley I test is ideal as a curing test for modern crosslinkers with methoxymethyl groups, because it shows very high formaldehyde values if the fabric is inadequately cured.

The European Committee for Standardization decided in 1993 to standardize only the LAW 112 and AATCC 112 methods, as EN ISO 14184 Parts 1 and 2 respectively. Both standards are now effective. The reason for standardizing only these two methods was their worldwide importance, especially LAW 112, which is the basis for the Finnish standard SFS 4996, for the method used by the German trade supervisory offices and, in particular, for the formaldehyde limits of Öko-Tex Standard 100. The Öko-Tex Standard 100 label is the textile finisher’s guarantee that, for example, the formaldehyde content of adult clothing fabrics worn next to the skin does not exceed the 75 ppm formaldehyde limit in the LAW 112 test. Furthermore, some retail organizations have laid down similar limits based on the same test method. From a medical point of view, the limits are arbitrarily defined values not based on medical research.

Formaldehyde test methods and their scope				
		EN ISO 14184		
		Part 1	Part 2	
	DIN 54260	LAW 112	AATCC 112 (BS 6806, pt. 3)	Shirley I (BS 6806, pt. 2)
Sample	*in* Na_2SO_3 solution	*in* water	*over* water	*in* water
Conditions	0 °C; pH 9.3–10.5	40 °C; 1 h	49 °C; 20 h	25 °C; 20 min
Analytical method	iodometric titration	colorimetric, acetylacetone	colorimetric, acetylacetone (H_2SO_4/chromotropic acid)	colorimetric, H_2SO_4/chromotropic acid
= $N–CH_2OCH_3$				✔
= $N–CH_2OH$		✔	✔	✔
CH_2O, $HO(CH_2O)_nH$, $HOCH_2OR$	✔	✔	✔	✔

Figure 1

2.4 Crosslinking agents for low-formaldehyde finishing

In the last 10 years, a revolution that no-one could have predicted has taken place in the field of crosslinking agents with regard to free formaldehyde content. In 1990, Fixapret CL was brought on to the market, followed by Fixapret CV, Fixapret ECO and finally Fixapret CM. Figure 2 gives a comparison of finishes produced with equal amounts of these crosslinkers (in terms of solids content) on cotton poplin. The finishes all showed approximately the same standard of finishing effects. Although they are modified derivatives of DMDHEU, the crosslinkers are very reactive even with magnesium chloride as the catalyst. It is usually sufficient when finishing cotton with these crosslinkers and magnesium chloride to double the reaction time or increase the curing temperature by 10 °C. Fixapret CL and Fixapret CM are very chlorine-resistant and are suitable for white goods. Fixapret CV is suitable where particularly low formaldehyde values in the Shirley I test are required, whilst the values obtained in the LAW 112 and AATCC 112 tests decrease in the order Fixapret CL, Fixapret CM, Fixapret ECO. Formaldehyde values below the Öko-Tex Standard 100 limit of 75 ppm can be reliably obtained only with Fixapret ECO.

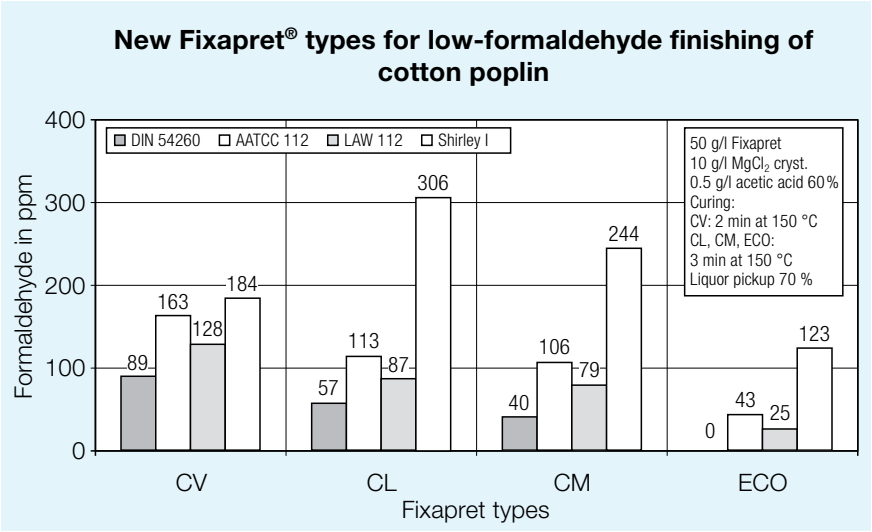


Figure 2

Regenerated cellulose is usually finished with reactive, non-methylated crosslinkers, such as Fixapret CNR Conc. (Fig. 3). Under the same application conditions, this product's more reactive successor, Fixapret CV, can improve the finishing effects and lower the formaldehyde values. Since viscose requires larger amounts of crosslinker than cotton does, the limits of Öko-Tex Standard 100 can generally only be met with Fixapret ECO, and recently also with Fixapret AP. Fixapret AP has the advantage that the carbon emissions are lower and that it allows even the more severe limits to be observed. The formaldehyde values shown by any crosslinker can be reduced with Condensol LF or Condensol FB. In the case of Fixapret ECO and Fixapret AP, these catalysts ensure reliable conformity to the 75 ppm limit in the plant.

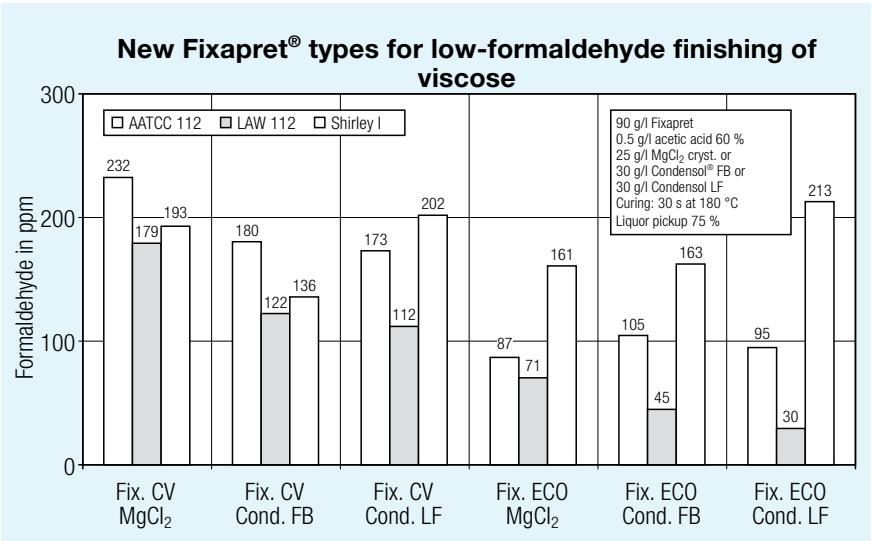


Figure 3

All the measurements shown here were carried out on samples that had been heat-sealed in plastic bags immediately after curing. The samples were tested as soon as possible, because even in sealed plastic bags the formaldehyde values can fall by up to 30 % within a month. In the case of unenclosed samples stored at 20 °C and a relative humidity of 65 %, the formaldehyde values measured in the LAW 112 and AATCC 112 tests can fall by almost 50 % in 20 h and by over 70 % in a week. The greatest drop in formaldehyde levels occurs in normal household washing. If Fixapret crosslinkers for low-formaldehyde finishing are used, the formaldehyde content according to the LAW 112, Shirley I and DIN 54260 tests falls to the detection limit in a single wash. The AATCC 112 values fall to just above the detection limit in one wash and reach the limit after further washes. This means that dry-crosslinked goods that have undergone an industrial wash can be regarded as practically formaldehyde-free in terms of Öko-Tex Standard 100.

As already mentioned, only the free formaldehyde content as measured by the LAW 112 test has any significance for the consumer. Therefore, with such small traces of releasable formaldehyde on the resin-finished textiles even before they are washed, allergic reactions even in sensitive persons are very unlikely.

2.5 Formaldehyde-free crosslinking agents

All real and supposed problems with the toxicity of formaldehyde in waste water and exhaust air, and in textiles when they are being worn, can be avoided with the formaldehyde-free crosslinker Fixapret NF. 1,3-dimethyl-4,5-dihydroxyethylene urea (DMeDHEU) is the only type of formaldehyde-free crosslinker to have gained acceptance in the world market. BASF offers Fixapret NF together with Condensol N, a system nearly as effective as traditional resin finishing with formaldehyde-based crosslinkers, and which on well neutralized fabric reduces to a minimum its disadvantages with regard to potential yellowing and odour formation. The application of Fixapret NF and Condensol N has become standard on particular qualities, e. g. to produce formaldehyde-free children's clothing, to reduce the strength losses in fabrics cured by the postcure process, and to obtain blends of cellulosic and synthetic fibres with a softer handle.

Figure 4 compares formaldehyde-free Fixapret NF and formaldehyde-based Fixapret CL. Fixapret NF produces finishes that are free of formaldehyde and have a much softer handle. The other effects are virtually the same.

Comparison of Fixapret® NF and Fixapret CL (PES/CO 67/33)

		0	1	2	3	4
Fixapret NF	g/l		80		80	
Fixapret CL	g/l			40		40
Condensol® N	g/l		30		30	
$MgCl_2$ cryst.	g/l			10		10
Acetic acid 60 %	g/l		0.5	0.5	0.5	0.5
FWA, stable to acid	g/l				5	5
Ultraphor® RN	g/l				4	4
Dry crease recovery angle (∑ warp + weft)	∢°	211	233	251	242	249
DP rating (wash 20 min at 60 °C, tumbler dried)	rating	2.5	3	3	3.3	3.3
AATCC 112	ppm	4	4	130	4	87
LAW 112	ppm	7	6	83	7	71
Degree of whiteness (Berger)		66.8	65.8	67.4	137.5	145.4

Liquor pickup 65 %, drying at 110 °C, curing 30 s at 180 °C,
0 = unfinished, FWA = fluorescent whitening agent

Figure 4

Because of the low strength losses obtained with Fixapret NF and Condensol N, this system is of particular interest for the postcure process. Compared with Fixapret ECO, the easycare effects are somewhat less favourable and the crease lines somewhat less permanent, but the tensile strength and abrasion resistance are much higher (Fig. 5). Moreover, there are no problems at all with formaldehyde from the ironing press or on the goods.

Postcure process with Fixapret® NF and Fixapret ECO				
		0	1	2
Fixapret NF	g/l		150	
Condensol® N	g/l		45	
Fixapret ECO	g/l			80
$MgCl_2$ cryst.	g/l			15
Siligen® VN	g/l		15	15
Basosoft® SWK	g/l		10	10
Dry crease recovery angle (∑ warp + weft) (1 x 20 min at 40 °C) (5 x 20 min at 40 °C)	 ∢° ∢°	 128 134	 245 199	 273 216
DP rating (5 washes 20 min at 40 °C, tumbler dried)	rating	2.5	3.5	3.5
Tensile strength 50 mm x 200 mm	N	519	421	217
Accelerotor weight loss (3 min, 3000 rpm)	%	5.7	7.6	24.8
AATCC 112 (before pressing)	ppm	4	4	134
AATCC 112	ppm	4	6	49
LAW 112	ppm	13	2	28
Liquor pickup 70 %, drying at 110 °C to 10 % residual moisture, steaming 5 s, pressing 20 s at 160 °C, curing 8 min at 150 °C, 0 = unfinished				

Figure 5

3 Catalysts

Every finishing recipe contains “catalysts”, which ensure that the reaction takes place within the 130 – 180 °C temperature range typically employed in the textile industry. The catalysts used in resin finishing are not real catalysts in the usual sense of the word, because the reaction rate is dependent on the amount of catalyst present.

3.1 General information on catalysts in resin finishing

Three classes of catalysts are distinguished in the dry crosslinking process commonly used today:

- Ammonium salts, e. g. ammonium chloride, sulfate and nitrate
- Metal salts, e. g. magnesium chloride, zinc nitrate, zinc chloride, aluminium sulfate and aluminium hydroxychloride
- Catalyst mixtures, e. g. magnesium chloride with added organic and inorganic acids or acid donors.

Ammonium salts used to be employed particularly with crosslinkers consisting of reaction products of urea and formaldehyde or melamine and formaldehyde, and in the finishing of regenerated cellulose. As the importance of these crosslinkers declined, so did the importance of the ammonium salt catalysts. Problems with liquor stability, shade changes and an occasional “fishy odour” also contributed to this decline.

The most commonly used catalyst is magnesium chloride. Although zinc nitrate is even more effective, associated yellowing problems and recent environmental concerns mean that it is now seldom used.

In many cases, magnesium chloride is supplemented with organic acids or inorganic Brønsted or Lewis acids.

Liquid catalyst mixtures are often available commercially. These are mostly based on magnesium chloride and organic acids, e. g. α-hydroxycarboxylic acids, or inorganic Lewis acids.

Since the catalysts used in resin finishing are not real catalysts, i. e. not quantity-independent, the application rate is calculated as a percentage of the amount of crosslinker. However, if the amount of crosslinker used is very large or very small, the amount of catalyst should not exceed a certain maximum or fall below a certain minimum.

Of general validity for the catalysis of resin finishing is the Arrhenius equation, which says that raising the curing temperature by 10 °C roughly halves the curing time, or that lowering it by 10 °C means the curing time must be doubled (Fig. 6). Of course this equation only applies approximately, because the heating-up characteristics of the textile material in the curing machine or on the stenter depend on the material's mass per unit area.

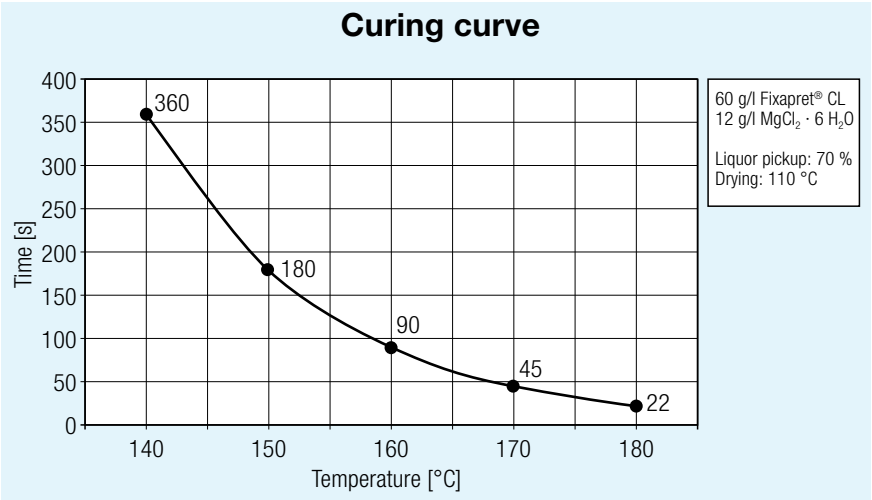


Figure 6

3.2 Condensol® types

BASF supplies three Condensol types:

- **Condensol FB**
- **Condensol LF**
- **Condensol N**

Condensol FB

A synergistic mixture of metal salts, Condensol FB is a highly effective catalyst for the resin finishing of textiles composed of cellulosic fibres and their blends with synthetic fibres. It allows traditional crosslinkers such as Fixapret CP Conc. or Fixapret CNR Conc. to be cured either in a much shorter time or at a much lower temperature than with conventional catalysts based on metal salts (Fig. 7).

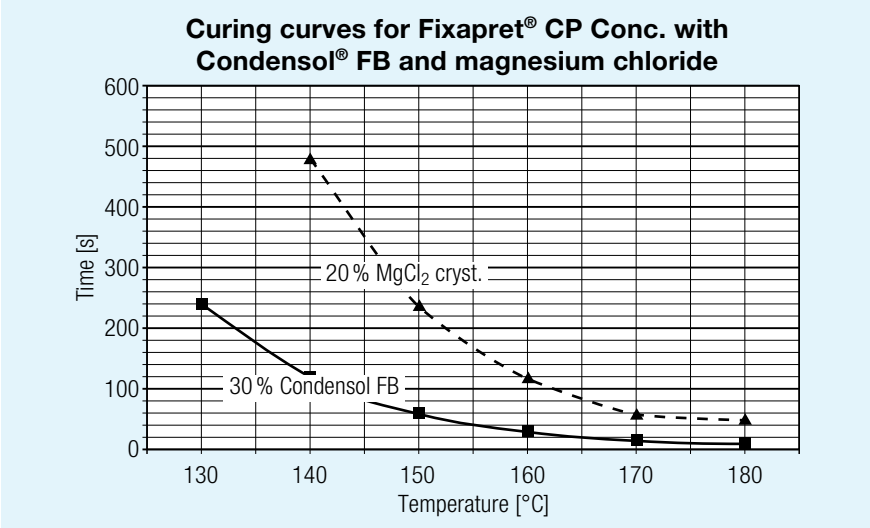


Figure 7

The shorter curing time in particular is often what first makes simultaneous drying and curing possible even on short stenters. On longer stenters, higher machine speeds can be achieved, considerably reducing drying costs. Another advantage of catalysing traditional crosslinkers with Condensol FB is the substantial reduction in formaldehyde values by about a third to a half. On blends of cellulosic and synthetic fibres dyed with disperse dyes, the milder curing conditions result in better wet and rub fastness properties, because less dye migrates to the fibre surface and dissolves in the finish film. A very important advantage is that Condensol FB overrides “buffered” conditions. When applied to alkaline fabric, in alkaline, hard or buffered process water, or with alkaline or buffered additives, Condensol FB effectively counteracts the buffer effect. Condensol FB can also be used as a partial replacement for magnesium chloride in recipes in which this is the catalyst, to improve the reactivity, shorten the curing time or lower the curing temperature.

Condensol FB is not as effective with reactive Fixapret crosslinkers for low-formaldehyde finishing as it is with Fixapret CP Conc. Nevertheless, it also increases the reactivity of these crosslinkers and counteracts the buffer effect of alkaline or buffering substances in the finishing liquor. It can be applied very successfully, alone or together with magnesium chloride, with all crosslinkers on fabrics composed of regenerated-cellulose fibres, since these fibres show hardly any loss in tensile strength due to resin finishing, even under drastic curing conditions, as long as suitable additives are used (Fig. 3).

Condensol LF

Condensol LF is a mixture of a metal salt and a polyamide solution. It is used as a catalyst for resin finishing textiles composed of cellulosic fibres and their blends with synthetic fibres. It gives much lower free-formaldehyde values in the LAW 112 and AATCC 112 tests (EN ISO 14184 Parts 1 and 2) than magnesium chloride does. The values obtained with crosslinkers for low-formaldehyde finishing usually fall below the formaldehyde limits of Öko-Tex Standard 100 (75 ppm in the LAW 112 test) without an afterwash. Condensol LF also makes the fabric soft, smooth and hydrophilic.

With the modern, reactive Fixapret types CL, CM, CV, ECO and AP, Condensol LF is ideal for further reducing the formaldehyde values obtained with magnesium chloride as the catalyst, without significantly affecting the reactivity (cf. Figs. 8 and 2). Thus LAW 112 formaldehyde values below 75 ppm are possible even with industrially resin-finished viscose spun-yarn fabrics, which require a large amount of crosslinker (Fig. 3).

Condensol® LF and Fixapret® types for low-formaldehyde finishing of cotton poplin

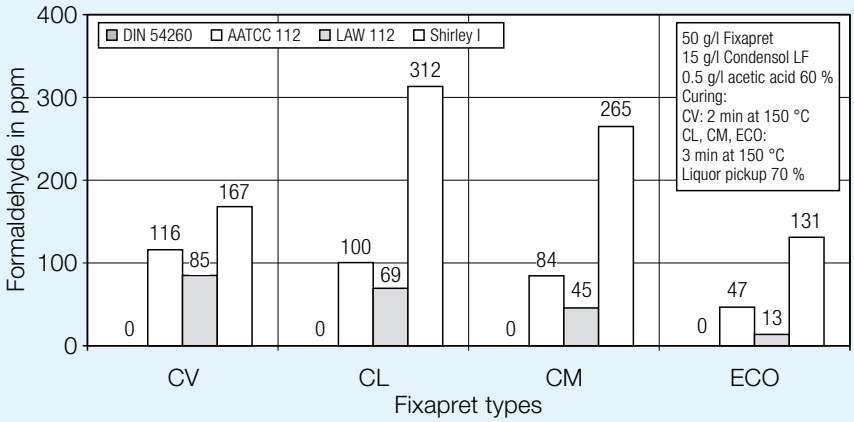


Figure 8

Condensol N

Condensol N is a synergetic mixture of inorganic salts. This catalyst is recommended **exclusively** for use with the formaldehyde-free crosslinker Fixapret NF for resin finishing textiles composed of cellulosic fibres and their blends with synthetic fibres. As already mentioned in discussing Fixapret NF, crosslinkers based on dimethyldihydroxyethylene urea tend to produce an odour when catalysed with magnesium chloride. This garlicky odour occurs especially when the fabric or finishing liquor has an alkaline pH or when the curing conditions are too extreme. Figure 4 shows that Fixapret NF, cured with Condensol N, produces only slightly poorer easy-care effects and a somewhat lower degree of whiteness than a formaldehyde-based crosslinker. By using Condensol N, the odour problem can be reliably avoided.

4 Additives

The term “additives” in resin finishing used to be restricted to those products that improved the effects or those that partly or completely compensated for the negative effects of crosslinking, such as a reduction in softness, hydrophilicity, abrasion resistance, tear strength, etc. Nowadays, all components added to resin-finishing recipes are called additives.

As a rule, the resistance of additives to washing and dry cleaning is greatly improved by applying them together with crosslinkers, because under these conditions cellulosic fibres do not swell to the same extent. This increase in permanence of the additives is particularly apparent with crosslinkers based on melamine and formaldehyde, which have very good crosslinking properties.

BASF’s range of finishing products includes the following additives for resin finishing:

- **Basosoft® types**
- **Helizarin® binders**
- **Perapret® types**
- **Persistol® types**
- **Siligen® types**
- **Texapret® types**

Most of these products are chemically very different, e. g.:

- Polymers based on acrylic monomers, vinyl monomers, siloxanes, amides, urethanes and ethylene
- Low-molecular substances such as fatty acid derivatives and quaternary ammonium compounds
- Mixtures of these substances

Polyacrylic esters

The main group of polymers used for finishing, pigment printing and pigment dyeing – because of the soft handle of the finishes obtained – are those based on acrylic esters ("polyacrylates") and containing a high proportion of acrylic acid butyl ester.

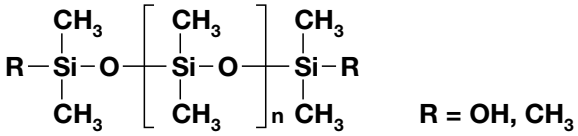
Dispersions of polyacrylic esters have the following general structure:

R1 = alkyl group of an alcohol
R2 = H or CH_3
X = CN, C_6H_5, CO-NH_2, CO-NH-CH_2-OH, etc.

Polysiloxanes

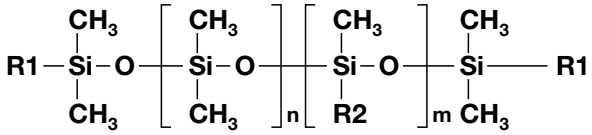
The second important group of polymers in finishing are the polysiloxanes. They owe their significance, despite the expense of the raw materials required for their manufacture, particularly to the special effects they impart to the finish, e.g. a very smooth handle and improved resin-finishing effects. Polysiloxanes have gained general acceptance since new, stable dispersions were developed, especially very stable, finely dispersed microemulsions, and dispersions of polysiloxanes containing amino groups. A further characteristic of crosslinkable polysiloxanes is the reactive hydroxy groups in the α and Ω positions, which are very important for the wash resistance of these finishing agents. The various polysiloxane types are shown below:

Polydimethylsiloxane and α, Ω-dihydroxypolydimethylsiloxane



R = OH, CH_3

Polydimethylsiloxane and α, Ω-dihydroxypolydimethylsiloxane, both containing amino groups



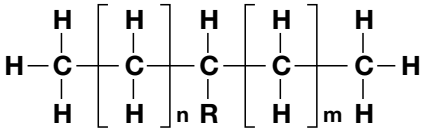
R1 = OH, CH_3
R2 = $(CH_2)_3$-NH-$(CH_2)_2$-NH_2, $(CH_2)_3$-NH_2

The polysiloxanes are supplied as aqueous emulsions. Sometimes these are "primary" dispersions, obtained by condensing emulsified tri- and tetra-oligomers. Usually, however, the silicone oil condensation product is dispersed with emulsifiers. Specialty products are the thermodynamically stable "microemulsions", which have an extremely small particle size (< 10 nm compared with a typical particle size of < 300 nm for milky emulsions) and are therefore clear.

Polyethylene waxes

Polyethylene wax (PE wax) dispersions are another important polymer group in finishing. They are partially oxidized polyethylenes known as waxes. In the oxidation process, a small number of hydrophilic groups are formed, allowing stable dispersions to be produced. Also available are "primary" polyethylene dispersions, manufactured by polymerizing emulsified ethylene and, for example, acrylic acid.

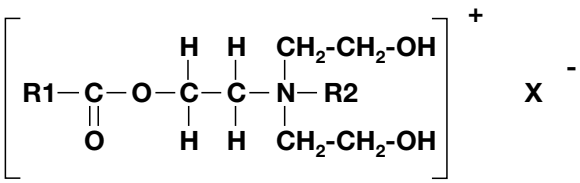
Polyethylene waxes have the following chemical structure:

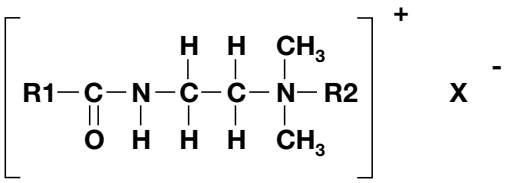


R = COOH

Softeners based on fatty acid derivatives

Fatty acid softeners occupy the largest volume share of the finishing market, particularly because they are also used in exhaust dyeing processes, e.g. in dyeing machines, washing machines, etc. There are anionic, cationic and nonionic softeners. The most important group, on account of their very good softening effect, are the cationic softeners, especially those that are fatty acid condensation products, despite some disadvantages such as yellowing at high temperatures and incompatibility with anionic finishing agents (e.g. fluorescent brighteners).





R1 = long-chain alkyl group
R2 = H, CH_3, benzyl group
X^- = Cl^-, $HCOO^-$, CH_3COO^-, $CH_3SO_4^-$

In the case of blended fabrics whose synthetic-fibre component has been dyed with disperse dyes, the rub and wet fastness properties may be affected by a tendency of the dyes to thermomigrate and dissolve in the additives, especially at high curing and fixation temperatures. In such cases preliminary trials are necessary.

4.1 Basosoft types

The Basosoft types are modified, silicone-free fatty acid derivatives. They impart a smooth, soft handle to textiles composed of cellulosic fibres and their blends with synthetic fibres. This effect compensates partly or completely, depending on the amount of crosslinker present, for the somewhat fuller, stiffer handle before the first wash of resin-finished fabrics compared with unfinished fabrics. After the first wash, the handle of resin-finished fabrics is in any case smoother and softer, even without softening, than that of unfinished fabrics. In addition, resin finishing increases the wash resistance of all additives.

The range of Basosoft types comprises the following products:

- **Basosoft D**
- **Basosoft EUK Conc.**
- **Basosoft SWK**

Basosoft D

Basosoft D is a fatty acid condensation product. It is a liquid, universally applicable cationic softener that is suitable for all fibre types and their blends. It produces a very soft handle and can be applied to loose stock, card sliver, tops, yarn or piece goods by a variety of methods, such as dipping, lick-roll application, spraying, etc. Indigo-dyed denim fabric that has been treated with Basosoft D yellows much less during storage.

Basosoft D can be applied in resin finishing, but as with all softeners of this type it must be remembered that at curing temperatures above 150 °C it can affect the degree of whiteness or the shade of pastel dyeings. Even small amounts give a very soft handle.

Basosoft EUK Conc.

Basosoft EUK Conc. is a fatty acid condensation product supplied in the form of flakes. It is a cationic softener that can be applied universally to all fibre types and their blends. It produces a very soft handle and can be applied to loose stock, card sliver, tops, yarn or piece goods by a variety of methods, such as dipping, lick-roll application, spraying, etc.

When applied in resin finishing, it can, like Basosoft D, affect the degree of whiteness or the shade of pastel dyeings if the finish is cured at a high temperature (above 150 °C). Even small amounts produce a very soft handle.

Basosoft SWK

Basosoft SWK is a nonionic, emulsified mixture of a fatty acid condensation product and a polyethylene wax. The weakly cationic dispersion is low-foaming, stable to shearing and free of paraffin wax and silicones. It is a smoothing and softening agent for all fibre types and their blends, has an antistatic effect, and is compatible with the usual fluorescent brighteners for cellulosic fibres. The product is an ideal additive for applying a raised finish. On knitted fabrics it improves the sewability. It can be applied to yarn and piece goods by padding and the exhaust process, e.g. in jet dyeing machines.

When applied as a softener in resin finishing, it imparts a soft, smooth handle to the fabric and increases the tear strength and abrasion resistance. On knitgoods it improves the sewability, which is adversely affected by resin finishing. Under typical curing conditions, it does not affect the degree of whiteness or shade.

4.2 Perapret types, Helizarin Binder FWT, Texapret AM

The Perapret types, Helizarin Binder FWT and Texapret AM are dispersions or solutions of film-forming polymers or polycondensates for various fields of application.

Chemically, they fall into four different groups:

- Solutions of polyvinyl acetate
- Dispersions of polyacrylic esters
- Dispersions of polyethylene
- Dispersions of polyurethane

BASF's range of these film-forming polymers comprises the following products:

- **Helizarin Binder FWT**
- **Perapret F 50**
- **Perapret HVN**
- **Perapret PE 40**
- **Perapret PU**
- **Perapret VA**
- **Texapret AM**

Helizarin Binder FWT

Helizarin Binder FWT is an aqueous dispersion of an acrylic ester copolymer. This thermally crosslinkable binder is suitable for pigment dyeing woven and knitted fabrics of all fibre types and fibre blends in pale to medium shades. The dyeings are fast to washing, dry cleaning (perchloroethylene) and ageing, and have a soft handle.

Combining pigment dyeing and resin finishing saves costs and simplifies and speeds up the finishing process. Moreover, the wash, dry-cleaning and rub fastness values are increased by the resin finish, and the tear strength, tensile strength and abrasion resistance are improved by the Helizarin Binder FWT. It is because of these last-named characteristics that Helizarin Binder FWT is also used in resin finishing.

Perapret F 50

Perapret F 50 is an anionic polyacrylate dispersion. It is a thermally crosslinkable finishing agent for woven and knitted fabrics composed of cellulosic fibres and their blends with synthetic fibres. Large application rates produce a full, stiff handle. The effects are largely resistant to washing and dry cleaning. It is suitable for gumming the selvedges of knitgoods.

In resin-finishing recipes, it is used as a filling and stiffening agent. Resin finishing increases the resistance of the effects to washing and dry cleaning. At the same time, Perapret F 50 improves the resin-finishing effects, so that the amount of crosslinker can be reduced. The same easycare effects are therefore accompanied by greater tear strength and abrasion resistance.

Perapret HVN

Perapret HVN is an anionic, thermally crosslinkable polyacrylate dispersion for finishing woven and knitted fabrics composed of cellulosic fibres and their blends with synthetic fibres. It reduces the tendency to pilling and improves the antislip properties of the seams.

As an additive in resin finishing, Perapret HVN, like Perapret F 50, increases the tear strength and abrasion resistance, but without significantly affecting the handle. Adding Perapret HVN to the resin-finishing liquor allows the amount of crosslinker to be reduced. Greater tear strength and abrasion resistance can therefore be achieved while maintaining the same standard of easycare effects.

Perapret PE 40

Perapret PE 40 is a primary, anionic polyethylene dispersion. It imparts a somewhat fuller, smoother handle to woven and knitted fabrics composed of cellulosic fibres and their blends with synthetic fibres.

When applied in resin finishing, it increases the tear strength and abrasion resistance and gives the fabric a somewhat fuller, smoother handle.

Perapret PU

Perapret PU is a film-forming, anionic polyurethane dispersion that is suitable for finishing and as a dyeing binder for woven and knitted fabrics of all fibre types. It increases the tear strength and abrasion resistance, reduces the tendency to pilling, and improves the antislip properties of the seams. It can be applied to cotton fabrics to obtain a chintz effect that is fast to washing and dry cleaning. As a binder in pigment dyeing, Perapret PU is characterized by a very high pigment binding capacity. It therefore yields deeper shades than conventional dyeing binders. Higher application rates result in a fuller handle. Applying Perapret PU together with Siligen FA effectively prevents dye migration and produces a soft handle, more even dyeings and more wash-resistant effects.

The easycare effects of crosslinking agents can be improved by applying them together with Perapret PU. Or the same effects can be obtained with a reduced amount of crosslinker, resulting in a higher tear strength and abrasion resistance and reducing the tendency of the fabric to pilling even after repeated washing.

Perapret VA

Perapret VA is a nonionic dispersion of polyvinyl acetate. It is a very high-yielding finishing agent for producing wash-resistant filling and stiffening effects on woven and knitted fabrics composed of cellulosic fibres and their blends with synthetic fibres. Larger amounts allow a very stiff handle to be obtained. It can be used for gumming the selvedges of knitgoods.

Combined with crosslinkers, it is suitable for finishing interlinings and workwear.

Texapret AM

Texapret AM is a nonionic polyacrylate solution. It is a finishing agent for producing filling and stiffening effects on woven and knitted fabrics composed of cellulosic fibres, wool and their blends with synthetic fibres. Larger amounts yield a stiff handle.

Applying it together with crosslinkers allows largely wash-resistant finishing effects to be achieved. It is particularly efficient on textiles of viscose spun yarn.

4.3 Persistol types

The Persistol types are traditional finishing agents for waterproofing textiles composed of natural and synthetic fibres and their blends. To be effective, the water-repellent product must be located at the surface of the textile material after finishing. Rapid drying and a high curing temperature, or wet-on-wet finishing, therefore lead to a particularly good water-repellent effect. The products do not contain any emulsifiers and should be stirred before being taken from the containers in which they are supplied. To obtain a good water-repellent effect, the untreated fabric must be as free as possible of surfactants – especially nonionic surfactants – and the finishing bath should not contain any wetting agents or surfactant-containing auxiliaries. The fabric should also be as closely woven and smooth as possible. When the Persistol types are applied to cotton together with resin-finishing agents, the cotton fibres swell less and the water-repellent effect is largely resistant to washing and dry cleaning.

BASF supplies the following water-repellents for resin finishing:

- **Persistol E**
- **Persistol HP**

Persistol E

Persistol E is a cationic, emulsifier-free paraffin wax emulsion containing zirconium. It is suitable for waterproofing textiles composed of natural and synthetic fibres and their blends. It can be applied by padding and by the exhaust process, i. e. also to wool. Calendering improves the effect.

Persistol E can be used together with crosslinking agents. This improves the water-repellent effect and makes it more resistant to washing and dry cleaning.

Persistol HP

Persistol HP is a cationic N-methylol compound substituted with a fatty acid. It is suitable for waterproofing woven fabrics composed of cellulosic fibres and their blends with synthetic fibres, and gives them a full, soft handle. In combination with oil-repellents it acts as an extender.

Persistol HP is usually applied together with resin-finishing agents, because like them it requires curing, and because on cotton they increase its resistance to washing and dry cleaning by reducing the swelling of the fibres. A better water-repellent effect is obtained with a zinc nitrate, zinc chloride or aluminium sulfate catalyst than with magnesium chloride, because the latter is hygroscopic. A particularly effective combination in terms of wash resistance is Persistol HP and Kaurit M 70 with zinc nitrate as the catalyst.

4.4 Siligen types

The Siligen types find application in resin finishing mainly as softening and smoothing agents. They vary greatly in chemical nature and include products based on long-chain aliphatics as well as dispersions of polysiloxanes and partially oxidized polyethylenes. The polysiloxanes and polyethylene waxes in particular are used not only to obtain the desired smooth handle, but also to improve the resin-finishing effects and compensate for the negative effects of resin finishing, such as reduction in abrasion resistance and tear strength. Because of their good wash resistance, these products are increasingly being used as additives in resin finishing, because the finishes are required to withstand a large number of washes.

BASF supplies the following Siligen types:

- **Siligen GL**
- **Siligen HS**
- **Siligen MSI**
- **Siligen PW**
- **Siligen SID**
- **Siligen SIA**
- **Siligen SIN**
- **Siligen SIO**
- **Siligen VN**

Siligen GL

Siligen GL is a nonionic mixture of fatty acid esters with an antifoam effect. It is recommended as a softener and smoothing agent for finishing woven and knitted fabrics composed of cellulosic fibres and their blends with synthetic fibres. The abrasion resistance and tear strength are increased. It is an ideal auxiliary for calender finishing designed to increase the lustre (chintz), and for raising and emerizing.

Siligen GL is recommended for resin-finishing recipes as an effective, low-priced smoothing agent that is readily combinable with other products because of its nonionic nature, and does not have a yellowing effect even at high curing temperatures. Applying it together with crosslinkers makes the smoothing effect and any lustre finish more resistant to washing and dry cleaning.

Siligen HS

Siligen HS, a cationic fatty acid ester, is a very effective softener with an antistatic effect that can be applied to all fibre types by both continuous and batch processes. The handle of textiles finished with this product is very soft.

Siligen HS is ideal as an additive in the resin finishing of woven and knitted fabrics, because it does not tend to yellow unless the curing temperature exceeds 160 – 170 °C.

Siligen MSI

Siligen MSI is an anionic, primary emulsion of a reactive α, ω-di-hydroxy-polydimethylsiloxane. It is a smoothing agent for woven and knitted fabrics composed of cellulosic fibres, wool, synthetic fibres and their blends with one another. It does not diminish the degree of whiteness even at high temperatures. In combination with anionic products and dispersions, such as fluorescent brighteners, polyacrylates, polyurethanes, etc., it is very stable to shearing. It improves the abrasion resistance and tear strength as well as the sewability.

Siligen MSI is recommended for resin finishing because it does not yellow at high temperatures. In addition, the product has very little effect on the rub and wet fastness properties of disperse dyeings on PES/CO woven and knitted fabrics, since it has a low solubility for any dye that thermomigrates to the surface of the fabric during curing.

Siligen PW

Siligen PW, which is based on a stearyl urea derivative, is a nonionic, reactive and therefore wash-resistant softener for woven and knitted fabrics composed of cellulosic fibres and their blends with synthetic fibres. It is slightly water-repellent and can therefore be combined with waterproofing agents without significantly reducing their water-repellent effect. The handle is somewhat dry and wool-like.

The softening, water-repellent effects of Siligen PW, like those of most finishing agents, can be made more wash-resistant by applying it together with resin-finishing agents. In addition, fixation of this reactive product is improved by curing it in the presence of a catalyst.

Siligen SIA

Siligen SIA, a slightly opaque emulsion of a nonionic, reactive polysiloxane containing amino groups, is a smoothing and softening agent for woven and knitted fabrics composed of cellulosic fibres, wool, synthetic fibres and their blends. Applied in slightly acid finishing liquors, it is very stable to shearing, increases the resilience, tear strength and abrasion resistance of wovens, and improves the elastic recovery and sewability of knitgoods. A very smooth handle is obtained. The finishing effects are particularly wash-resistant. If PES/CO fabrics dyed with disperse dyes are finished with

Siligen SIA, it has very little effect on the rub and wet fastness properties, since it has a low solubility for any dye that thermomigrates to the surface of the fabric during curing.

The high wash resistance of Siligen SIA makes it very suitable for combination with crosslinkers. The smooth handle and improved resin-finishing effects, such as crease recovery, smoothness after washing, tear strength and abrasion resistance, are retained after many washes. It is therefore an ideal product for high-grade finishes.

Siligen SID

Siligen SID is a very finely dispersed (particle size <10 nm), almost clear microemulsion of a polysiloxane with amino groups. It is suitable as a slightly hydrophilic softening and smoothing agent for woven and knitted fabrics composed of cellulosic fibres and their blends with synthetic fibres. It is very stable to shearing, produces a very soft, smooth, supple handle, and improves the abrasion resistance, tear strength and sewability.

When applied together with crosslinkers, it improves the resin-finishing effects, such as crease recovery and smoothness. At the same time, it compensates for the negative effects of resin finishing on the abrasion resistance, tear strength and sewability.

Siligen SIN

Siligen SIN is a concentrated, very finely dispersed (particle size <10 nm), clear microemulsion of a polysiloxane with amino groups. It is suitable as a slightly hydrophilic softening and smoothing agent for woven and knitted fabrics composed of cellulosic fibres and their blends with synthetic fibres. It is very stable to shearing, produces a very soft, smooth, supple handle, and improves the abrasion resistance, tear strength and sewability.

When applied together with crosslinkers, it improves the resin-finishing effects, such as crease recovery and smoothness, making them largely wash-resistant. At the same time, it compensates for the negative effects of resin finishing on the abrasion resistance, tear strength and sewability.

Siligen SIO

Siligen SIO, a slightly opaque emulsion of a nonionic, reactive polysiloxane containing amino groups, is a smoothing and softening agent for woven and knitted fabrics composed of cellulosic fibres, wool, synthetic fibres and their blends. Applied in slightly acid finishing liquors, it is stable to shearing, increases the resilience, tear strength and abrasion resistance of wovens, and improves the elastic recovery and sewability of knitgoods. A very smooth, soft handle is obtained. The finishing effects are particularly wash-resistant. If PES/CO fabrics dyed with disperse dyes are finished with Siligen SIA, it has very little effect on the rub and wet fastness properties, since it has a low solubility for any dye that thermomigrates to the surface of the fabric during curing.

The high wash resistance of Siligen SIA makes it very suitable for combination with crosslinkers. The smooth handle and improved resin-finishing effects, such as crease recovery, smoothness after washing, tear strength and abrasion resistance, are retained after many washes. It is therefore an ideal product for high-grade finishes.

Siligen VN

Siligen VN is a nonionic secondary emulsion of a partially oxidized, high-melting polyethylene. It is a smoothing agent that increases the tear strength and abrasion resistance and improves the sewability and elastic recovery of knitgoods. Because of its high melting point, the finishing effects of this polyethylene wax are very wash-resistant.

Siligen VN's permanence makes it an ideal additive in resin finishing, because the improvement in crease recovery, smoothness, tear strength and abrasion resistance is retained after many washes. In the case of cotton knitgoods, it is particularly effective in offsetting the adverse effect of resin finishing on the sewability.

4.5 Influence of some additives on resin-finishing effects

As already mentioned several times, some additives have an influence on the resin-finishing effects. When applied together with crosslinking agents, they can both improve the resin-finishing effects, such as crease recovery and smoothness after washing, and partly or completely compensate for the negative effects of crosslinking, such as poorer sewability and reduced abrasion resistance and tear strength, etc. Particularly effective in this respect are the Siligen types SIA, SID, SIN, SIO and VN, which should therefore never be absent from any recipe for high-grade resin finishing. Figures 9 – 15 below illustrate the effect of several important additives on the crease recovery, abrasion resistance, tear strength, tensile strength and sewability before, and in some cases after, washing.

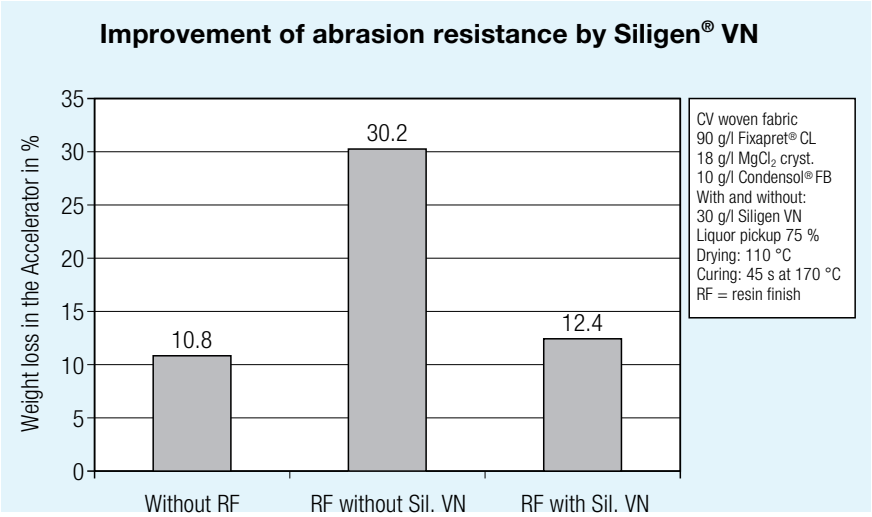


Figure 9

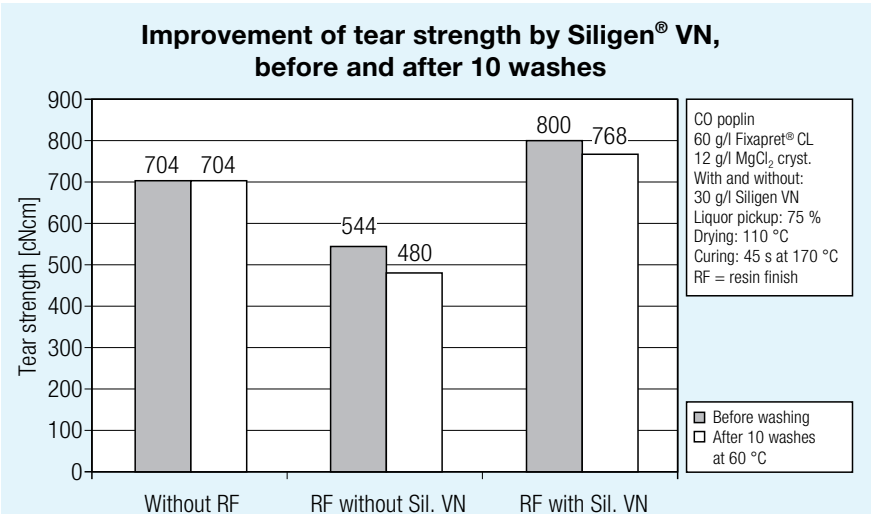


Figure 10

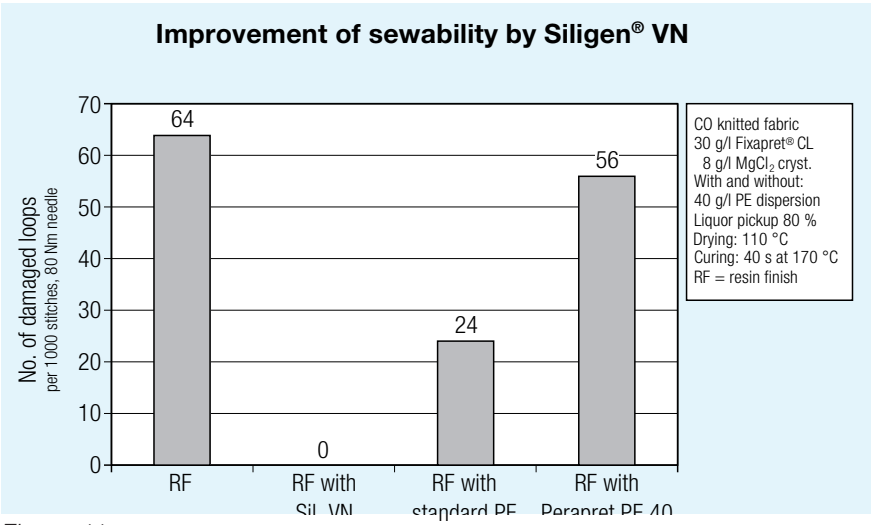


Figure 11

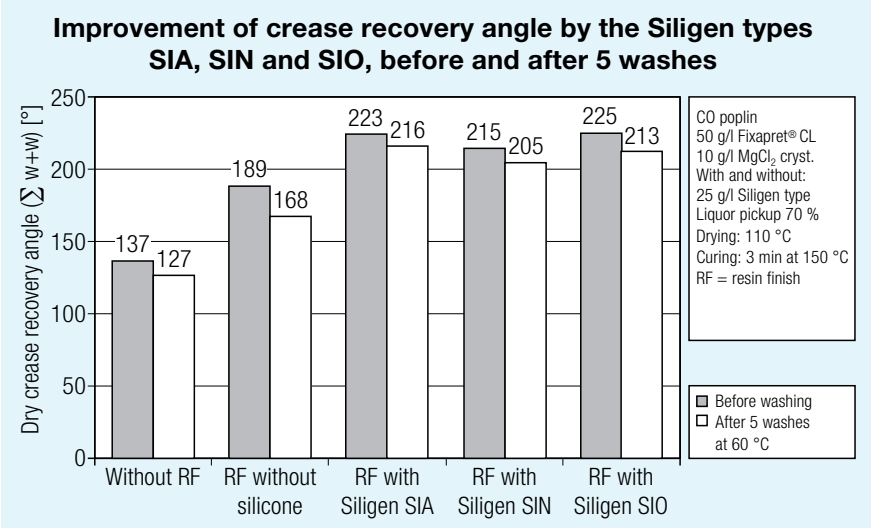


Figure 12

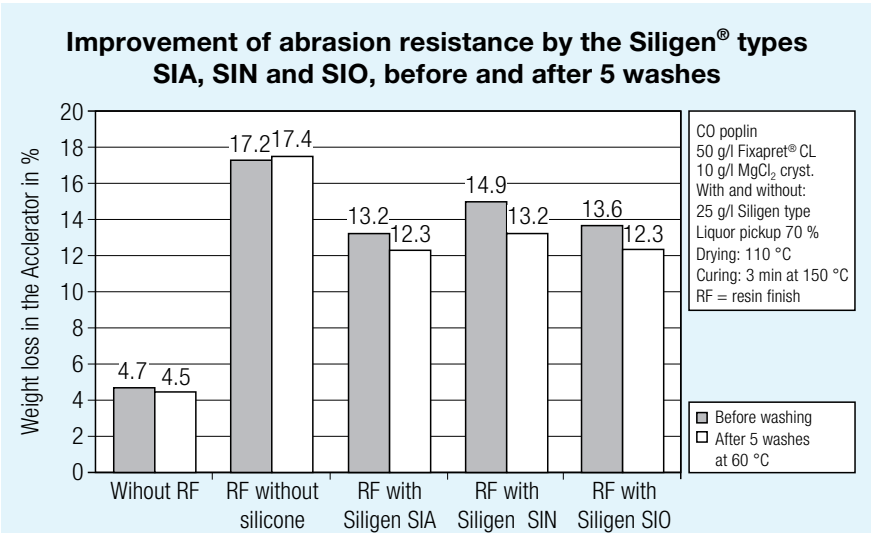


Figure 13

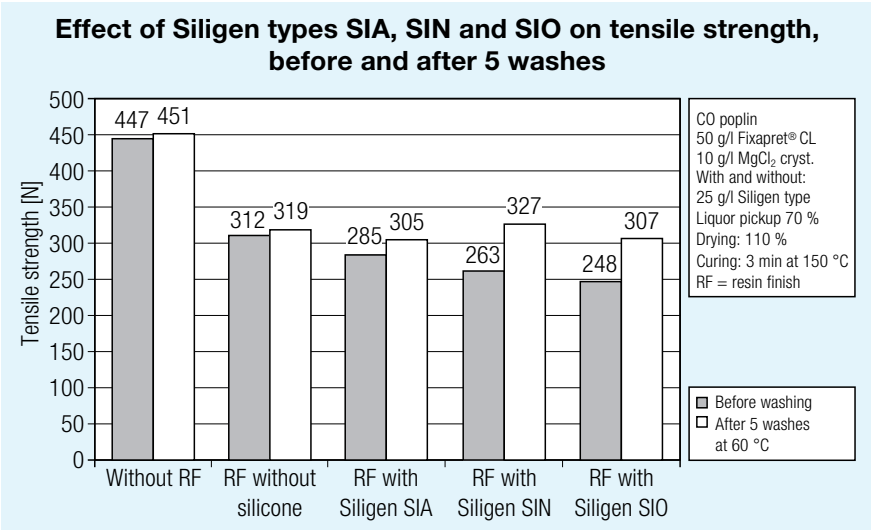


Figure 14

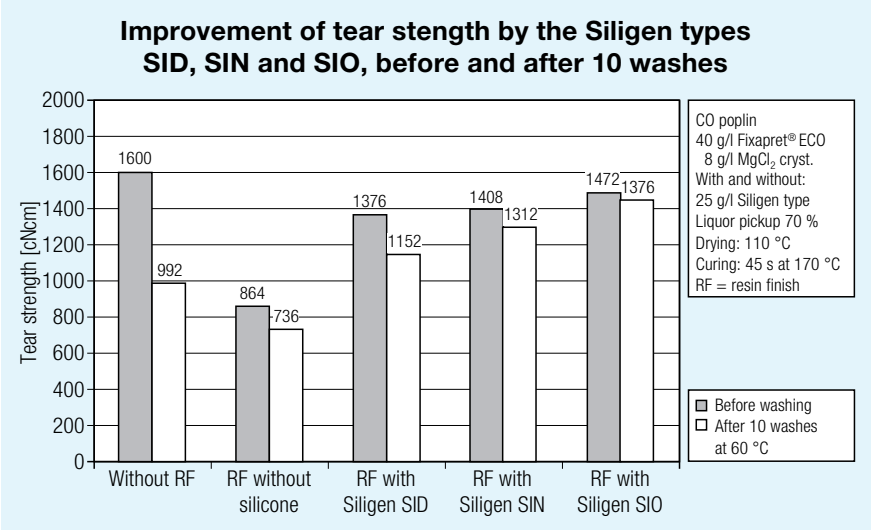


Figure 15

5 Surfactants in resin finishing

As with most textile finishing processes, every resin-finishing recipe contains surfactants, added either specially or indirectly via additives.

5.1 General information on surfactants in resin finishing

Wetting agents are essential to every resin-finishing recipe, because they ensure rapid wetting of the textile material during impregnation and even distribution of the liquor throughout the material. This applies especially to pigment-printed fabrics. In resin finishing, nonionic surfactants with both emulsifying and wetting properties are used. A good wetting agent should have little tendency to foam.

In recipes for resin finishing or for combined resin finishing and pigment dyeing, emulsifiers have the task of preventing liquor components from precipitating. The recipe components must meet exacting requirements because the shearing forces in the padder are extremely high. If the recipe includes additives of slightly opposite charge, or dispersions that are insufficiently stabilized or not stabilized for a particular application, then these may separate out in the strongly saline (catalyst) liquors used in resin finishing. The liquor pH also plays an important role in the stability. This problem can usually be avoided, or at least greatly reduced, with emulsifiers. In many cases, emulsifiers are already required in the manufacture of the additives, e. g. emulsion polymerisates and other dispersions. Moreover, additives are often specially stabilized with emulsifiers for resin finishing.

For resin finishing or for combined resin finishing and pigment dyeing, 1 – 2 g/l of a concentrated surfactant is recommended, depending on the stabilization effect required.

An enormous variety of surfactants exists. Only the most important group of nonionic surfactants for resin finishing, the ethoxylates, will be discussed here. The general formula of an ethoxylate is:

$R–O–[CH_2CH_2O]_n–H$

R = long-chain fatty alcohol or alkylphenol group

5.2 BASF surfactants

The following BASF surfactants are recommended for finishing and pigment dyeing:

- **Kieralon® JET-B Conc.**
- **Laventin® LNB**
- **Nekanil® LN**

Kieralon JET-B Conc.

Kieralon JET-B Conc. is a clear to opaque, low-viscosity, liquid mixture of nonionic surfactants. It is recommended in textile processing as a universally applicable, low-foaming wetting agent and detergent. It is free of alkylphenol ethoxylates (APEO-free).

Kieralon JET-B Conc. has proved to be a very effective, non-foaming wetting agent in resin finishing and in combined resin finishing and pigment dyeing. In liquors containing surfactants it can even be used as a foam suppressant. Owing to its emulsifying properties, it has a very good stabilizing effect on finishing liquors containing oppositely charged additives or other products. The stability of dispersions in resin-finishing liquors is strongly influenced by the catalyst. Here, too, Kieralon JET-B Conc. counteracts any tendency to form precipitates or roller deposits. These stabilizing properties and its foam-suppressant effect make the product ideal for combined pigment dyeing and resin finishing.

Because of its outstanding properties, Kieralon JET-B Conc. is generally recommended by BASF for resin-finishing recipes.

Laventin LNB

Laventin LNB is a highly concentrated ethoxylated fatty alcohol. This low-foaming nonionic surfactant is a clear, low-viscosity liquid with an excellent wetting and detergent effect. It is free of alkylphenol ethoxylates (APEO-free).

Since Laventin LNB must be carefully dissolved and is not as low-foaming as Kieralon JET-B Conc., it is no longer so widely used in finishing and pigment-dyeing recipes.

Nekanil LN

Nekanil LN is an anhydrous, slightly viscous, yellowish alkylphenol ethoxylate that is readily soluble in water. It is a low-foaming nonionic surfactant with an outstanding wetting and detergent effect at low temperature.

Despite its excellent wetting and emulsifying properties, it has lost importance in finishing and pigment-dyeing recipes because it is not as low-foaming as Kieralon JET-B Conc. Since Nekanil LN is an alkylphenol ethoxylate, in some countries it may no longer be used in the textile industry, because its breakdown products are toxic to fish.

6 Combined resin finishing and pigment dyeing

BASF was one of the pioneers of pigment printing and dyeing in the 1950s. These processes involve fixing pigments on fabric in a manner that is independent of the fibre by means of film-forming polymers. Because of the simple application technique, the relatively low processing costs and the good fastness properties obtained, pigment printing and dyeing continue to grow in worldwide importance. Pigment dyeing is carried out in the same machines as resin finishing, i. e. a liquor containing pigment preparations and a polymer is padded on to the fabric, which is then dried and cured, usually on a stenter. Since resin finishing and pigment dyeing involve the same machinery, it is logical when applying them to woven or knitted fabrics composed of cellulosic and synthetic fibres to combine the two processes in one step.

6.1 General information on combined resin finishing and pigment dyeing

The simultaneous application of resin finishing and pigment dyeing to fabrics composed of cellulosic and synthetic fibres combines the advantages of both processes.

Combining resin finishing and pigment dyeing:

- is economic, because drying and curing need to be carried out only once, allowing large cost savings in handling, energy, chemicals and water
- is simple, because the fabric only needs to be padded, dried and cured, and no aftertreatment is required
- is environmentally sound, because there is very little contamination of waste water or exhaust air
- results in very reproducible dyeings
- makes brilliant shades possible
- further increases the good fastness values obtained in resin finishing, so that not only are the pigments extremely lightfast, but they are fast to washing, dry cleaning and rubbing as well
- produces easycare fabrics, i. e. virtually crease-proof fabrics that do not shrink on washing and need very little ironing
- is extremely versatile, because it means that woven or knitted fabrics composed of cellulosic and other fibres do not need to be dyed with different types of dyes in a number of separate steps, but can be dyed in a single step merely with pigments and a dyeing binder, regardless of the kinds of fibres present
- allows a finishing plant to carry out dyeings on existing machinery, without the need for additional investments

6.2 Products for pigment dyeing

Recipes for one-bath resin finishing and pigment dyeing consist of the following products:

- *Crosslinker:* modified DMDHEU
- *Catalyst:* usually magnesium chloride
- *Softening and smoothing agent:* e. g. nonionic types
- *Dyeing binder:* film-forming polymer based on polyacrylic esters or polyurethanes
- *Pigment preparations:* preparations of organic or inorganic pigments
- *Antifoam:* mixture of nonionic alkoxylates
- *Antimigrant:* nonionic ethoxylation product
- *Emulsifier:* nonionic alkoxylates that stabilize the dye liquor, prevent roller deposits and wet the fabric.

BASF has the following range of products for combined pigment dyeing and resin finishing:

- **Fixapret types and selected softening and smoothing agents of the resin-finishing range (see sections 2 – 5), e. g. Fixapret types ECO, CL, etc., Siligen VN, Basosoft SWK, Siligen MSI**
- **Helizarin pigment preparations for pigment dyeing**
- **Helizarin Binder FWT, Perapret PU**
- **Kieralon JET-B Conc. (see 5.2)**
- **Siligen FA**
- **Vitexol® PFA**

Helizarin pigment preparations for pigment dyeing

BASF supplies highly coloured pigment preparations of organic and inorganic pigments in many hues for pigment dyeing. The pigment preparations, ranging from brilliant to muted, can be mixed with one another to obtain the desired shades. Even at low concentration, the dyeings usually have a high standard of light fastness and conform to Öko-Tex Standard 100.

Helizarin Binder FWT

Helizarin Binder FWT is an aqueous dispersion of an acrylic ester copolymer. The thermally crosslinkable binder is suitable for pigment dyeing woven and knitted fabrics of all fibre types and fibre blends in light to medium shades. The dyeings are fast to washing, dry cleaning (perchloroethylene) and ageing, and have a soft handle.

Combining pigment dyeing and resin finishing saves costs and simplifies and speeds up processing. In addition, the wash, dry-cleaning and rub fastness values are increased by resin finishing, and the tear strength, tensile strength and abrasion resistance are improved by Helizarin Binder FWT. Owing to these latter properties, Helizarin Binder FWT is also used in resin finishing alone (see p. 19).

Perapret PU

Perapret PU is a film-forming, anionic polyurethane dispersion used as a dyeing binder on woven and knitted fabrics of all fibre types. In pigment dyeing, Perapret PU is characterized by a very high pigment binding capacity. It therefore allows dyeings to be produced in deeper shades than conventional dyeing binders do. Increasing the application rate results in a fuller fabric handle.

Applying pigment dyeing and resin finishing together saves costs and simplifies and speeds up processing. In addition, the wash, dry-cleaning and rub fastness values are increased by resin finishing, and the tear strength, tensile strength and abrasion resistance are improved by Perapret PU. Owing to these latter properties, Perapret PU is also used as an additive in resin finishing alone (see. p. 20).

Siligen FA

Siligen FA is a nonionic ethoxylation product with softening and hydrophilizing properties. It is recommended as an antimigrant in pigment dyeing and one-bath pigment dyeing and finishing. Much more even dyeings are obtained with greater dye penetration. Since there is better distribution of the dyeing binder and therefore of the pigment, a higher standard of fastness is achieved. In addition, a softer handle is obtained.

For these reasons, Siligen FA has also proved its effectiveness as an antimigrant in pigment-free finishes with the above dyeing binders or with other dispersions of film-forming polymers such as polyacrylic esters and polyurethanes. It results in a softer handle and improves the desired finishing effects, such as reduced pilling.

Vitexol PFA

Vitexol PFA is a nonionic mixture of alkoxylates recommended as an antifoam in pigment dyeing and in combined pigment dyeing and finishing. It is free of silicones and phosphoric acid esters. By applying Vitexol PFA, foam formation in the pad-box and hence mottling of the fabric are largely prevented. Furthermore, it effectively prevents deposits from forming on the pad rollers.

6.3 Recipes for one-bath resin finishing and pigment dyeing

Recipe for pale shades:

0.1	–	3	g/l	Helizarin® pigment
		2	g/l	Kieralon® JET B Conc.
		4	g/l	Vitexol® PFA
10	–	20	g/l	Siligen® FA
50	–	100	g/l	Helizarin Binder FWT
40	–	60	g/l	Fixapret® ECO
8	–	12	g/l	magnesium chloride cryst.

Liquor pickup: 65 % (as low as possible)
Drying: 100 – 120 °C
Curing: 30 – 40 s at 170 °C (according to the fabric)

Recipe for dark shades

The same recipe as for Helizarin Binder FWT plus:
up to approx. 50 g/l Helizarin pigment
up to approx. 100 g/l Perapret PU

Note

The information submitted in this publication is based on our current knowledge and experience. In view of the many factors that may affect processing and application, these data do not relieve processors of the responsibility of carrying out their own tests and experiments; neither do they imply any legally binding assurance of certain properties or of suitability for a specific purpose. It is the responsibility of those to whom we supply our products to ensure that any proprietary rights and existing laws and legislation are observed.

Printed in Germany

BASF Aktiengesellschaft **Regional Marketing Europe** Ludwigshafen – Germany	Tel.: (49) 621 60 4 54 81 Tel.: (49) 621 60 0	Fax: (49) 621 60 4 58 87
BASF Aktiengesellschaft **Regional Marketing Asia** Singapore	Tel.: (65) 432 34 00	Fax: (65) 432 34 34
BASF Corporation **Regional Marketing NAFTA** Charlotte, NC – USA	Tel.: (1) 704 392 43 13	Fax: (1) 704 393 36 49
BASF S.A. **Regional Marketing South America** São Bernardo do Campo/São Paulo – Brazil	Tel.: (55) 11 751 22 33	Fax: (55) 11 751 69 89
BASF Aktiengesellschaft **Regional Marketing Eastern Europe, Africa and Westasia** Ludwigshafen – Germany	Tel.: (49) 621 60 4 57 78 Tel.: (49) 621 60 0	Fax: (49) 621 60 2 04 22

Colorants and Finishing Products

BASF