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# A Study of the Surface and Total Cyclic Trimer Content of Polyester Filament Yarns and Staple Fibres Available in South Africa

by

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# A STUDY OF THE SURFACE AND TOTAL CYCLIC TRIMER CONTENT OF POLYESTER FILAMENT YARNS AND STAPLE FIBRES AVAILABLE IN SOUTH AFRICA

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## ABSTRACT

*The total amount of methylene chloride extractable matter, the total cyclic trimer content and the surface trimer content of 23 commercial polyester staple and filament samples were determined. The total methylene chloride extractable matter content of the samples varied from 1,4 to 4,6 per cent, (average value 2,6 per cent), the total cyclic trimer content varied from about 0,7 to 1,7 per cent (average value 1,2 per cent) and the surface trimer content varied from <0,01 to 0,25 per cent (average value 0,10 per cent).*

*The effect of various treatments on the surface trimer content of polyester was also investigated. Heat-setting and curing treatments had little effect on the surface trimer content, but autoclave-steaming for 30 minutes at 130°C increased it considerably. Dyeing in the presence of carrier had the largest effect on the surface trimer content of the polyester and in some cases increased it to values as high as 0,50 per cent.*

## INTRODUCTION

The role of oligomers in the processing of synthetics has been thoroughly investigated in recent years. It is well-known that the migration of oligomers, especially under high temperature conditions, can cause problems in the processing of polyester fibres. Oligomers comprise the low molecular weight homologues of polymers, where the basic polymer unit is repeated a limited number of times only[1]. Typical examples are the dimers, trimers, tetramers, etc. Oligomers can occur in linear or cyclic forms.

Linear oligomer:

$$\left[ -O-CH_2-CH_2-O-\overset{\overset{O}{\|}}{C}-C_6H_4-\underset{\underset{O}{\|}}{C}- \right]_n$$

Cyclic oligomer:

$$\left[ -O-CH_2-CH_2-O-\overset{\overset{O}{\|}}{C}-C_6H_4-\underset{\underset{O}{\|}}{C}- \right]_n \text{ (cyclic)}$$

Oligomers are normally formed during the polymerisation process. In the case of polyester the formed polymer contains a relatively low concentration of oligomers. These oligomers do not interfere with the spinning process and consequently they are not removed from the polymer prior to spinning. In the case of technical Nylon 6, however, the polycondensation product contains up to 12 *per cent* low molecular weight products which interfere with the spinning process and which must therefore be removed from the polymer[2].

Various values have been reported for the oligomer content of polyester fibres. Some authors[2,3] reported values as high as 4,0 *per cent*, but nowadays it is generally accepted that the oligomer content of polyester is about 1,5 to 2,0 *per cent*[4]. Polyester fibres contain several different linear and cyclic oligomers but the main constituent is the *cyclic trimer* cyclo-tris-ethylene-glycol terephthalate ($c(GT)_3$). Most authors[4] state that the cyclic trimer forms about 90 to 95 *per cent* of the total oligomer content of polyester, but some others have quoted values as low as 70 to 80 *per cent*[5,6]. It is generally accepted, however, that the cyclic trimer is the main cause of many of the problems experienced in the processing of polyester fibres or yarns.

Oligomers are found at the nozzle head during spinning and may also be encountered during draw-twisting and texturing[7]. During dyeing oligomers may be extracted from the fibre, followed by a redeposition on the fabric and the walls of the dyeing apparatus[2,8,9]. Some authors state, however, that the oligomers are not extracted from the fibre but migrate from the inside and recrystallise directly on the fibre surface[10]. In some cases problems may arise during rewinding and knitting due to the shedding of the oligomers from the fibre surface onto the equipment. Oligomers can have an adverse effect on the friction of the fibres and it has been stated that the fibres can melt as a result of the heat which is developed.

The concentration of oligomers on the fibre surface will have a significant effect on the processing characteristics of the polyester. The concentration of oligomers on the surface of the fibre is normally very low and values as low as 0,004 *per cent* have been quoted[11]. In general the surface oligomer concentration seems to be less than 0,02 *per cent*, based on the fibre mass[3,4,6]. Various authors have proposed maximum tolerance values for the surface oligomer content of polyester. Nitschke[4], for example, stated that polyester with a cyclic trimer content less than 0,2 *per cent* would not give any problems during processing. Valk and co-workers[10] and Dugal *et al*[5] on the other hand, claimed that problems could be encountered when the surface oligomer content was higher than 0,1 *per cent*.

Apart from the oligomers which originally may be present on the fibre surface, it is also known that oligomers inside the fibre migrate to the surface during heat treatments, thereby increasing the oligomer concentration on the fibre surface. Treatments such as texturing and heat-setting increase the surface oligomer concentration only slightly to about 0,01 to 0,02 *per cent*[3,12]. In general, heat treatments below 120°C seem to have practically no effect on the surface oligomer concentration. Treatments above 120°C, however, especially in the presence of water, can increase the surface oligomer concentration considerably. Valk *et al*[10] showed that steaming for 30 minutes at 130°C increased the surface oligomer concentration of polyester from 0,05 *per cent* to 0,13 *per cent*. Lang and Makart[6] found that autoclave-setting for 30 minutes at 130°C increased the surface oligomer concentration to about 0,16 *per cent*.

The effect of dyeing on the surface oligomer content of polyester is well-known. Dyeing at temperatures lower than 120°C in general has little effect on the surface oligomers. When the dyeing temperature exceeds 120°C, however, the surface oligomer content of the fibres increases significantly. Valk *et al*[10], Vernazza[13] and others[4,14] showed that values higher than 0,25 *per cent* can be obtained after dyeing at 130°C. When dyeing is carried out in the presence of carriers the surface oligomer content can increase even further to values higher than 0,50 *per cent*. In fact, the presence of carriers can result in excessive surface oligomer formation, even when dyeing is carried out at temperatures as low as 110°C. Valk *et al*[10], for example, showed that the surface oligomer content of polyester dyed at 110°C, varied from 0,13 to 0,58 *per cent*, depending on the quantity and type of carrier used. Another aspect which should be referred to is the observation by Reinert and co-workers[15] that polyester which had been pretreated or scoured in hot perchloroethylene showed a higher tendency towards oligomer migration during dyeing than polyester scoured in cold perchloroethylene or hot water. They showed that there was a good correlation between the quantity of perchloroethylene retained by the polyester and the surface trimer content after dyeing.

Von der Eltz *et al*[7] claimed that the chances were slight that a universal remedy to problems caused by cyclic trimers would be found in the immediate future. However, they were of the opinion that the continued improvement of dyeing machines and the dropping of the dye-liquor as hot as possible would make it easier to deal with such problems. Baker[9] also recommended the "hot drop" technique as a possible remedy to the trimer problem. Other authors, such as Dugal *et al*[14] claimed, however, that the "hot drop" technique or the use of dispersing agents did not reduce the surface trimer content of polyester. This is in agreement with the findings of Valk *et al*[10], who showed that the trimer on the fibre surface after dyeing was not the result of a redeposition of trimer from the dyeing liquor, but was the product which migrated from the fibre inside to the outside. Vernazza[13] agreed with this observation, but pointed out that the "hot drop" technique reduced the build-up of trimer in the dye-bath. Levy[16] pointed out that the "hot drop" technique could result in trimer condensation in the heat exchange unit, since the liquor started cooling down there prior to its discharge. Recently Stein and co-workers[17] claimed that the oligomer problem in dyeing could be solved by the correct scouring or pretreatment of the polyester prior to dyeing.

Research on polyester oligomers, especially with reference to problems encountered in processing and dyeing, is of a very recent nature. In fact, most publications on this topic have been published since 1974. Little is known about the cyclic trimer content of the various polyesters used in South Africa and it was considered important to determine these values and to study the effect of certain treatments on the migration of the trimer.

## EXPERIMENTAL

**Materials:**

Commercially available polyester filament yarns and staple fibres were used. All chemicals used were of laboratory grade quality.

**Treatments:**

Heat treatments were carried out in a laboratory oven. Autoclave-steaming was carried out in an Andrews autosetter. Blank dyeing trials and treatments with perchloroethylene were performed in the containers of a Linitest apparatus.

**Tests:**

The *surface trimer content* of the polyester was determined by shaking the samples in dioxane for 10 minutes at room temperature. Valk *et al*[2] and Kusch[3] showed that this treatment removed only the surface oligomers from the fibres. The *total trimer content* of the samples was determined by soxhlet extraction with methylene chloride for one hour. Lang and Makart[6] showed that this treatment removed all the oligomers from the polyester. After extraction, the dioxane and methylene chloride extracts were diluted to a specific volume, from which aliquots were transferred to a thin layer chromatography plate. (Silica Gel F254). Reference solutions containing known amounts of pure *cyclic trimer* were prepared and aliquots from these were also placed on the thin layer plate. The plate was then transferred to a chromatography tank containing benzene/dioxane (10:1) as eluent. When the eluent front had moved about 10 cm the plate was removed from the tank, dried and then viewed in ultraviolet light (254 nm). The cyclic trimer content of the samples was determined by comparison with the standards[3]. The thin layer plates showed that the solvent extracts also contained some other oligomers. However, these oligomers formed a small part of the total oligomer content of the polyester and were ignored in this investigation.

The cyclic trimer which was used as reference sample was extracted from polyester, and was recrystallised and purified according to the method described by Lang and Makart[6]. It had a melting point of 312° to 316°C, which is in agreement with values quoted in the literature.

Scanning electron microscope photographs were taken by the Electron Microscope Section, CSIR, at a magnification of 1 000.

## RESULTS AND DISCUSSION

The total amount of methylene chloride extractable matter for various polyester samples is given in Table I. It can be seen that the values ranged from 1,4 *per cent* to 4,6 *per cent*, but most varied, however, from 1,8 to 3,3 *per cent*. This is in agreement with the results of Kusch[18], who analysed 24 commercial yarns used

**TABLE I**
**THE TOTAL METHYLENE CHLORIDE EXTRACTABLE MATTER CONTENT, TOTAL TRIMER CONTENT AND SURFACE TRIMER CONTENT OF DIFFERENT COMMERCIAL POLYESTER SAMPLES**

| Sample | | Total Methylene Chloride extractable Content (%) | Total Trimer Content (%) | Surface Trimer Content (%) |
|---|---|---|---|---|
| No. | Description | | | |
| 1 | 1/85 f30 Nat. circ* | 1,7 | 1,4 | 0,01 |
| 2 | 1/85 f15 Nat. circ. | 3,3 | 0,8 | 0,06 |
| 3 | 1/110 f30 Nat. circ. | 2,9 | 0,9 | 0,19 |
| 4 | 1/150 f44 Nat. circ. | 2,7 | 1,1 | 0,25 |
| 5 | 1/167 f30 Nat. circ. | 2,7 | 1,1 | 0,19 |
| 6 | 1/167 f30 Nat. circ. | 2,6 | 1,3 | 0,01 |
| 7 | 1/167 f30 Nat. circ. | 4,6 | 1,4 | 0,01 |
| 8 | 1/167 f30 Dyed circ. | 3,9 | 0,4** | 0,25 |
| 9 | 1/167 f30 Dyed circ. | 2,1 | 0,6** | 0,19 |
| 10 | 1/167 f30 Nat. tri* | 2,7 | 1,1 | 0,19 |
| 11 | 1/167 f30 Nat. tri | 3,3 | 1,2 | 0,01 |
| 12 | 1/167 f30 Dyed tri | 2,5 | 0,9 | 0,19 |
| 13 | 1/200 f60 Nat. circ. | 2,4 | 1,1 | 0,25 |
| 14 | 2/167 f30 Nat. | 4,2 | 0,7 | 0,25 |
| 15 | 2/167 f30 Dyed | 3,7 | 0,7 | 0,25 |
| 16 | Polyester tops | 1,9 | 1,7 | < 0,01 |
| 17 | Polyester tops | 1,8 | 1,6 | < 0,01 |
| 18 | Short staple polyester | 1,8 | 1,4 | 0,01 |
| 19 | Low pilling polyester | 1,6 | 1,3 | 0,01 |
| 20 | Low pilling polyester | 1,4 | 1,1 | 0,01 |
| 21 | Normal polyester | 1,9 | 1,3 | < 0,01 |
| 22 | Low pilling polyester | 1,8 | 1,1 | < 0,01 |
| 23 | Low pilling polyester | 1,8 | 1,4 | < 0,01 |
| Average | | 2,6 | 1,2 | 0,10 |

*circ = circular cross-section
*tri = trilobal cross-section

**could not be determined accurately, due to the interference from some dissolved dyestuff.

in Germany, and found that only one yarn contained less than 1,5 *per cent* solvent extractable matter, while only two contained more than 4,0 *per cent*. Not all the methylene chloride extractable matter, however, comprised lubricants or spinning oils. Table I also shows that a relatively high percentage of the total extract consisted of the cyclic trimer of polyester (on average about 42 *per cent*). This indicates that the use of solvent extraction methods for determining the quantity of spinning oils and other additives on polyester will in most cases lead to erroneous (too high) values, due to the removal of polyester oligomers from the fibre. The average total cyclic trimer content of the polyester which is currently being used in South Africa seems to be about 1,2 *per cent*. The individual results varied from about 0,7 to 1,7 *per cent*. This is normal and in agreement with the results of Kusch[18], Dugal *et al*[5] and Lang and Makart[6].

Table I also gives the surface cyclic trimer content of the various polyester samples. Fairly low values were obtained in most cases. Twelve samples contained

**TABLE II**
**THE EFFECT OF VARIOUS HEAT AND STEAM TREATMENTS ON THE SURFACE TRIMER CONTENT OF POLYESTER**

| Sample | | Treatment | Surface trimer content (%) |
|---|---|---|---|
| No. | Description | | |
| 16. | Polyester tops | Heat 160°C, 3 min. | <0,01 |
| | | Heat 170°C, 3 min. | <0,01 |
| | | Heat 180°C, 3 min. | 0,01 |
| 17. | Polyester tops | Heat 160°C, 3 min. | <0,01 |
| | | Heat 170°C, 3 min. | <0,01 |
| | | Heat 180°C, 3 min. | 0,01 |
| 17. | Polyester tops | 10% ®Fixapret CP + 1 % zinc nitrate, 170°C, 3 min. | <0,01 |
| 17. | Polyester tops | 10% ®Fixapret CP + 1 % ammonium chloride, 170°C, 3 min. | <0,01 |
| 16. | Polyester tops | Autoclave steam. 110°C, 30 min. | 0,01 |
| | | Autoclave steam. 120°C, 30 min. | 0,03 |
| | | Autoclave steam. 130°C, 30 min. | 0,13 |
| 17. | Polyester tops | Autoclave steam. 110°C, 30 min. | 0,01 |
| | | Autoclave steam. 120°C, 30 min. | 0,03 |
| | | Autoclave steam. 130°C, 30 min. | 0,08 |

0,01 *per cent* (or less) surface trimer, seven samples contained from 0,01 to 0,20 *per cent* trimer, while only four samples contained more than 0,20 *per cent* trimer. Various authors claimed that a value of 0,1 *per cent* is the maximum tolerance value for the surface trimer content, but more recently Nitschke[4] stated that this value is too low and that problems could be encountered with the processing or dyeing of polyester containing more than about 0,2 *per cent* surface trimer. It is interesting to note that the dyed samples generally contained about 0,20 *per cent*, or more, surface trimer. However, some undyed samples also had very high surface trimer contents.

The effect of various heat and steam treatments on the surface trimer content of some of the polyester samples is shown in Table II. It can be seen that heat treatments for three minutes at temperatures up to 180°C had very little effect on the surface trimer contents of the samples. This is in general agreement with results published by other workers[11]. It was furthermore considered important to establish what the effect of various conditions, used for the curing of aminoplast resins on certain cotton/polyester blends, would be on the surface trimer content of the polyester. These results are also given in Table II. It can be seen that the treatment of polyester with a DMDHEU type resin and various catalysts did not increase the surface trimer content to any extent.

Table II furthermore shows the effect of autoclave steaming treatments on the surface trimer content of some polyester samples. Steaming at 110°C for 30 minutes had little effect on the surface trimer content, but when the steaming temperature was increased to 130°C, there was a significant (approximately ten-fold) increase in surface trimer content. This is in agreement with the results of other workers[6,10,12]. Lang and Makart[6], for example, observed a ten-fold increase in the surface oligomer content of polyester yarns, and found that the polyester had a surface trimer content of about 0,16 to 0,18 *per cent* after autoclave steaming.

The effect of certain scouring techniques on the surface trimer content of polyester during subsequent dyeing treatments is shown in Table III. Normally it would be expected that scouring pretreatments, especially with solvents such as perchloroethylene, would remove most of the surface trimer as well as a fair percentage of the oligomers in the fibre. Scouring pretreatments should therefore reduce the problem of oligomer migration during dyeing. In practice, however, this is not always the case. For example, Reinert and co-workers[15] found that polyester which had been pretreated in hot perchloroethylene showed a higher tendency towards oligomer migration during dyeing than polyester scoured in cold perchloroethylene or hot water. The effect of different scouring techniques on the surface trimer content of various polyester samples is shown in Table III. It can be seen that a scour in perchloroethylene removed practically all the surface trimer from the fibre. Scouring in water, however, was less effective and did not remove much of the surface trimer, as can be seen in the case of the samples containing a high percentage of surface trimer.

## TABLE III
## THE EFFECT OF VARIOUS SCOURING PRETREATMENTS ON THE SURFACE TRIMER CONTENT OF POLYESTER BEFORE AND AFTER DYEING

| Sample | | Treatment | Surface Trimer Content (%) | | | |
|---|---|---|---|---|---|---|
| | | | Before Scouring | After Scouring Before Dyeing | After Blank Dyeing | |
| No. | Description | | | | 100°C | 130°C |
| 16 | Polyester tops | Perchloroethylene: 120°C, 15 min. Dried 120°C, 5 min. | <0,01 | 0,02 | <0,01 | 0,01 |
| | | Perchloroethylene: 75°C, 15 min. Dried 120°C, 5 min. | | <0,01 | 0,02 | – |
| | | Perchloroethylene: 120°C, 15 min. Dried 95°C, 5 min. | | <0,01 | – | 0,01 |
| | | Water. 75°C, 15 min. Dried 120°C, 5 min. | | <0,01 | <0,01 | 0,02 |
| | | Water. 75°C, 15 min. Dried 95°C, 5 min. | | – | – | 0,02 |
| 17 | Polyester tops | Perchloroethylene: 120°C, 15 min. Dried 120°C, 5 min. | <0,01 | 0,01 | <0,01 | 0,01 |
| | | Perchloroethylene: 75°C, 15 min. Dried 120°C, 5 min. | | <0,01 | 0,02 | – |
| | | Perchloroethylene: 120°C, 15 min. Dried 95°C, 5 min. | | <0,01 | – | <0,01 |
| | | Water, 75°C, 15 min. Dried 120°C, 5 min. | | <0,01 | <0,01 | 0,04 |
| | | Water 75°C, 15 min. Dried 95°C. 5 min. | | – | – | 0,04 |
| 3 | 1/110 f30 Nat circ | Perchloroethylene: 120°C, 15 min. Dried 120°C, 5 min. | 0,19 | 0,01 | <0,01 | 0,01 |
| | | Perchloroethylene: 75°C, 15 min. Dried 120°C, 5 min. | | <0,01 | <0,01 | – |
| | | Perchloroethylene: 120°C, 15 min. Dried 95°C, 5 min. | | <0,01 | – | <0,01 |
| | | Water. 75°C, 15 min. Dried 120°C, 5 min. | | 0,13 | 0,25 | 0,44 |
| | | Water. 75°C, 15 min. Dried 95°C, 5 min. | | – | – | 0,31 |
| 4 | 1/150 f44 Nat circ | Perchloroethylene: 120°C, 15 min. Dried 120°C, 5 min. | 0,25 | 0,02 | 0,01 | 0,04 |
| | | Perchloroethylene: 75°C, 15 min. Dried 120°C, 5 min. | | 0,02 | <0,01 | – |
| | | Perchloroethylene: 120°C, 15 min. Dried 95°C, 5 min. | | <0,01 | – | 0,01 |
| | | Water. 75°C, 15 min. Dried 120°C, 5 min. | | 0,25 | 0,31 | 0,50 |
| | | Water. 75°C, 15 min. Dried 95°C, 5 min. | | – | – | 0,38 |

Table III furthermore shows that the surface trimer content of the samples with a low *original* surface trimer content increased only slightly after dyeing. The samples scoured in water had a slightly higher surface trimer content after dyeing than the samples scoured in perchloroethylene. When polyester samples with high *original* surface trimer contents were considered it was found that the surface trimer content of the samples scoured in perchloroethylene was still low after dyeing, while that of samples scoured in water increased considerably. No explanation can be offered at this stage for this observation, which is not in agreement with the findings of Reinert *et al*[15]. It is possible, however, that the fibres used in this investigation did not retain any perchloroethylene after drying, which could enhance the migration of trimer during dyeing. Another possibility could be the removal of all the surface trimer and most of the trimer inside the fibre by the perchloroethylene treatment. This matter will be investigated in more detail in future.

**TABLE IV**
**THE EFFECT OF CARRIER ON THE SURFACE TRIMER CONTENT OF POLYESTER DYED AT VARIOUS TEMPERATURES**

| Polyester | Dyeing Temperature | Carrier | Surface Trimer Content (%) |
|---|---|---|---|
| 1/150 f44 Nat. circ. | Undyed | - | 0,25 |
| 1/150 f44 Nat. circ. | 100°C | None | 0,19 |
| 1/150 f44 Nat. circ. | 110°C | None | 0,19 |
| 1/150 f44 Nat. circ. | 130°C | None | 0,19 |
| 1/150 f44 Nat. circ. | 100°C | ®Invalon | 0,25 |
| 1/150 f44 Nat. circ. | 110°C | Invalon | 0,25 |
| 1/150 f44 Nat. circ. | 130°C | Invalon | 0,44 |
| 1/150 f44 Nat. circ. | 100°C | ®Remol P | 0,25 |
| 1/150 f44 Nat. circ. | 110°C | Remol P | 0,25 |
| 1/150 f44 Nat. circ. | 130°C | Remol P | 0,50 |
| 1/167 f 30 Nat. circ. | Undyed | - | 0,01 |
| 1/167 f 30 Nat. circ. | 130°C | None | 0,06 |
| 1/167 f 30 Nat. circ. | 130°C | Remol P | 0,25 |

Liquor ratio 1 : 60
90 minutes
2 g/ℓ carrier

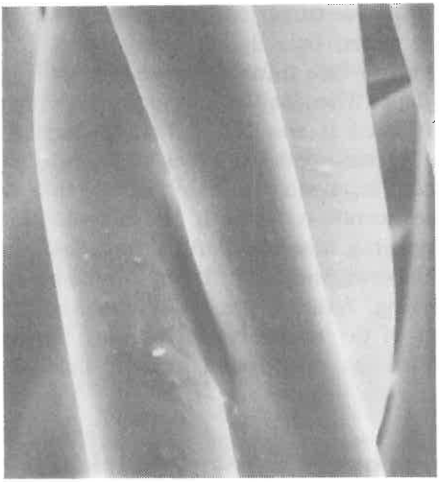

FIGURE 1
Scanning electron microscope photograph of untreated polyester with a *low* surface trimer content (Sample No. 7, 1/167 f30 Nat. circ).

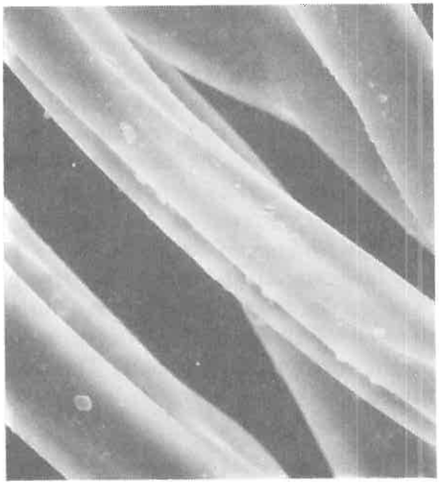

FIGURE 2
Scanning electron microscope photograph of untreated polyester with a *high* surface trimer content (Sample No. 4, 1/150 f44 Nat. circ).

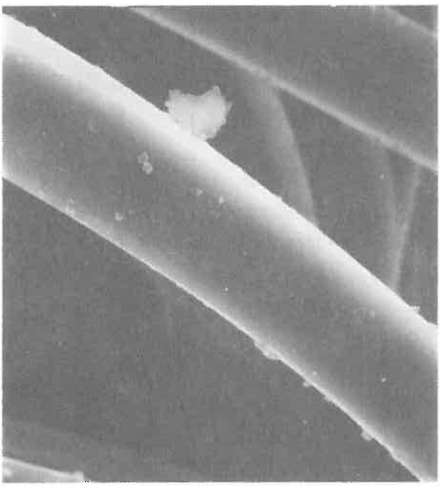

FIGURE 3
Scanning electron microscope photograph of sample No. 7 after blank dyeing in the presence of carrier.

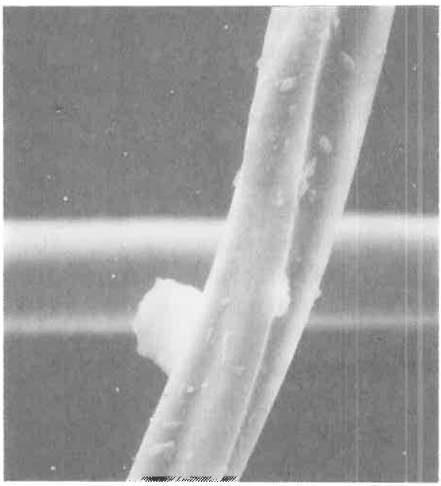

FIGURE 4
Scanning electron microscope photograph of Sample No. 4 after blank dyeing in the presence of carrier.

The effect of dyeing at different temperatures in the absence and presence of various carriers on the surface trimer content of polyester is shown in Table IV. It can be seen that, in this specific case, dyeing temperature had little effect on the surface trimer content. when dyeing was carried out in the absence of the carrier. In the case of the sample with the high surface trimer content, the values were, in fact, slightly lower after dyeing than before dyeing, probably due to the removal of some of the surface trimer during dyeing. The presence of carrier had little effect on the surface trimer content of the samples when blank dyeings were carried out at relatively low temperatures, such as 100°C and 110°C. When the dyeing temperature was increased to 130°C, however, there was a significant increase in the surface trimer content of the polyester. These results are in agreement with the findings of Valk *et al*[10], who found values as high as 0,58 *per cent* surface trimer after dyeing, and Nitschke[4], who reported values as high as 0,60 *per cent.*

Furthermore it can be seen that there was little difference between the two carriers used during the dyeing trials. This is in agreement with the findings of Valk *et al*[10], who stated that most carriers increased the surface trimer content of polyester significantly. Out of a range tested they found only one carrier which had a relatively small effect on the surface oligomer content. This carrier was of a type which did not act as a strong swelling agent.

Finally some scanning electron microscope photographs were taken of the undyed samples and the samples dyed at 130°C in the presence of Remol P. These photographs are shown in Figures 1 to 4. It can be seen that the surfaces of the two undyed samples differed considerably. The surface of the fibres with the low surface trimer content (Figure 1) was fairly smooth and even, while that of the fibres with the high surface trimer content (Figure 2) clearly showed the presence of numerous small protrusions. Dyeing resulted in the migration of oligomers from the inside of the fibres to the surface, and in both cases increased the quantity and size of the protrusions (Figures 3 and 4). Some of the protrusions were almost of the same diameter as the fibres, but were firmly bound to the fibres and could not be removed by rinsing in hot water.

## SUMMARY AND CONCLUSIONS

The total amount of methylene chloride extractable matter, the total trimer content and the surface trimer content of 23 commercial polyester staple and filament samples were determined. On average the samples contained 2,6 *per cent* methylene chloride extractable matter. About 42 *per cent* of this extract comprised the cyclic trimer cyclo-tris-ethylene-glycol terephthalate (c $(GT)_3$). This level (1,2 *per cent*) is normal and is in agreement with results published by other workers. The individual results varied from about 0,7 to 1,7 *per cent* total cyclic trimer. The average surface cyclic trimer content of the samples was fairly low. Twelve samples contained about 0,01 *per cent* cyclic trimer, or less, and four samples contained more than 0,20 *per cent*. Such a high surface trimer content could lead to processing and dyeing problems under certain conditions.

The effect of various heat and steam treatments on the surface trimer content of the polyester was investigated. Heat treatments for three minutes at temperatures up to 180°C had practically no effect on the surface trimer content, but autoclave-steaming for 30 minutes at 130°C increased it considerably.

The effect of certain scouring pretreatments on the surface trimer content of the fibres after dyeing was also studied. Scouring in perchloroethylene removed most of the surface trimer, but scouring in water had little effect on the surface trimer content. When the scoured samples were dyed the surface trimer content of the samples scoured in perchloroethylene remained fairly low, while that of the samples scoured in water increased considerably.

Furthermore the effect of carriers on the surface trimer content of some of the samples was investigated. Blank dyeings were carried out in the absence and presence of various carriers. Dyeing in the absence of carrier had little effect on the surface trimer content, while the presence of carrier increased the surface trimer content of polyester significantly, when dyeing was performed at 130°C. In some cases the surface trimer content of the polyester samples increased to about 0,50 *per cent* when dyeing was carried out at 130°C in the presence of 2 g/ℓ carrier.

Finally some scanning electron microscope photographs of some of the samples were taken. The increase in surface oligomer concentration as a result of carrier dyeing could clearly be seen in these photographs.

## THE USE OF PROPRIETARY NAMES

The fact that products with proprietary names (® denotes registered trade marks) have been used in this report does not in any way imply that SAWTRI recommends them or that there are not substitutes which may be of equal value or even better. ®Remol is the trade mark of Messrs Hoechst Ltd and ®Fixapret and ®Invalon those of Messrs Ciba Geigy.


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