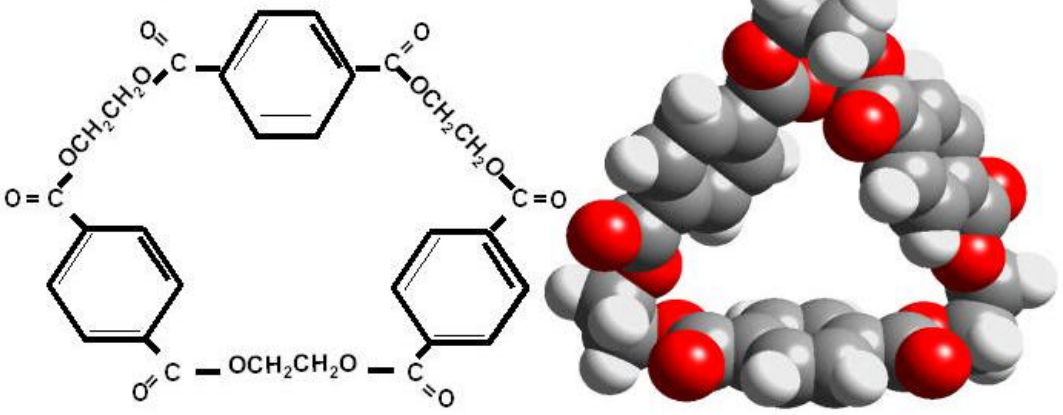
Problems with oligomer in dyeing polyester yarns and fabrics.

Typically polyester fibres contain between 1.5 and 3.5% by mass of low M_r esters, the principal oligomer being cyclic tris(ethylene terephthalate) with smaller quantities of a dimer, pentamer as well as traces of other compounds.

Polyester Oligomer

cyclic tris (ethylene terephthalate)
between 1.5% and 3.5% by mass



Problems caused by oligomer deposits

- Spinning characteristics impaired
- Reduced liquor flow through package of yarn because spindle perforations become blocked, and deposits on pump cause improper pump pressure.
- Presence can cause nucleation and growth of dye crystals or agglomeration of dye particles and hence dye spots, unlevelness, and poor fastness.
- Deposits on machine guides at winding or twisting cause high tensions and increased friction on the yarn, leading to poor package build and end breaks.
- White powdery deposits cause dulling of the yarn/fabric especially in dark shades, black, navy etc.
- Variation in rate of temperature rise due to deposits on heat exchanger
- Need for more frequent cleaning of dyeing and winding machines, hence increased down time and lower efficiency.

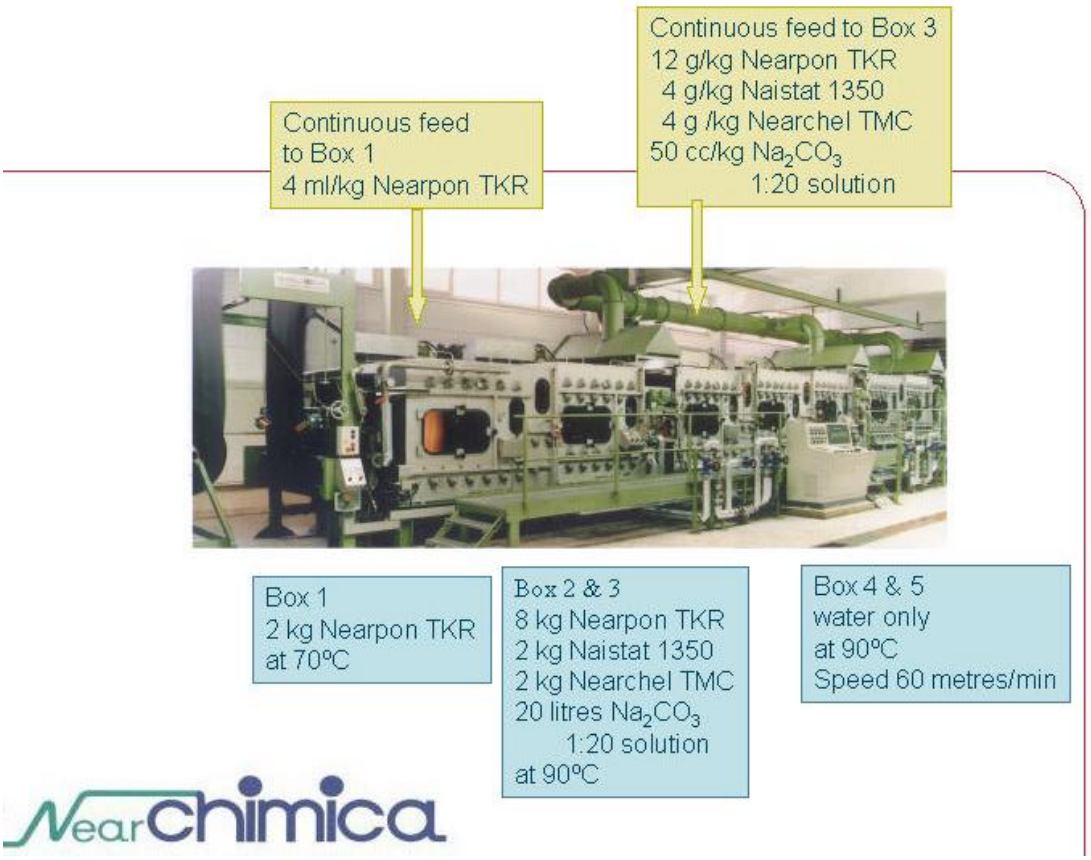
In some markets we have recently seen evidence of rather higher concentrations of oligomer, particularly in yarn processing where the deposition of crystalline oligomers as a white powder on the fibre surface and on the mechanism of winding machinery causes problems.

Cyclic trimers migrate from PET fibre during dyeing and steam setting and, to a lesser extent, during dry heat setting. The amount of oligomer migrating to the fibre surface increases with increase in temperature, and with prolonged dyeing time, therefore the liberation of oligomers during dyeing can be minimised by lowering the dyeing temperature from 130° to 120° C and by using the shortest possible dyeing time.

We have found that there is no simple, one-product solution to the problem of oligomer deposits, but rather that a series of precautions can help.

1. Preparation

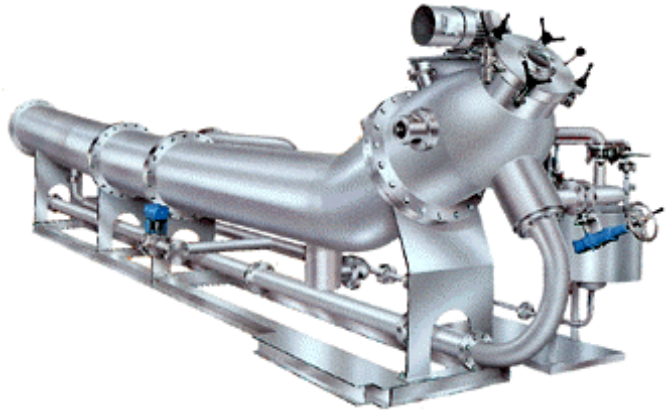
Often polyester yarn and fabric may be dyed with no preparation at all. However, when problems with oligomer deposits are being experienced we have found that a scouring treatment before dyeing can remove some oligomers from the outset.
Customers routinely preparing woven polyester fabric on an open width washing range, mainly to remove size, have noted also a marked reduction in oligomers present after scouring with a Nearchimica recipe.



Solvent extractions from polyester fabrics before and after scouring show the reduction in oligomer content after scouring with Nearpon TKR , Naistat 1350 and Nearchel TMC.

The table below shows results from production on a Babcock washing range

Fabric	Pet Ether extract	Methyl Ethyl Ketone extract	Total
100% polyester 1 greige	0.85% ester lubricant	0.8% PES oligomer & size	1.65%
1 scoured	0.2 %	0.35%	0.55%
100% polyester 2 greige	0.8% ester lubricant	1.2% PES oligomer & size	2.0%
2 scoured	0.2 %	0.6%	0.8%
100% polyester 3 greige	0.75% ester lubricant	1.35% PES oligomer & size	2.1%
3 scoured	0.3%	0.6%	0.9%
100% polyester 4 greige	0.95% fatty acid esters	0.85% PES oligomers	1.8%
4 scoured	0.25%	0.3 % includes surfactant	0.55%
100% polyester 5 greige	0.15% ester lubricant	1.15% PES oligomers	1.3%
5 scoured	zero	0.45% includes surfactant	0.45%
100% polyester 6 greige	0.1% synthetic esters	0.35% PES oligomers	0.45%
6 scoured	Zero	0.15%	0.15%
100% polyester 7 greige	0.9% esters & silicone	0.67% oligomers & acrylic	1.57%
7 scoured	0.06%	0.13% only trace acrylic size	0.19%
100% polyester 8 greige	0.8% natural esters	0.8% Polyester size & olig	1.6%
8 scoured	0.08 %	0.26% size absent	0.34%

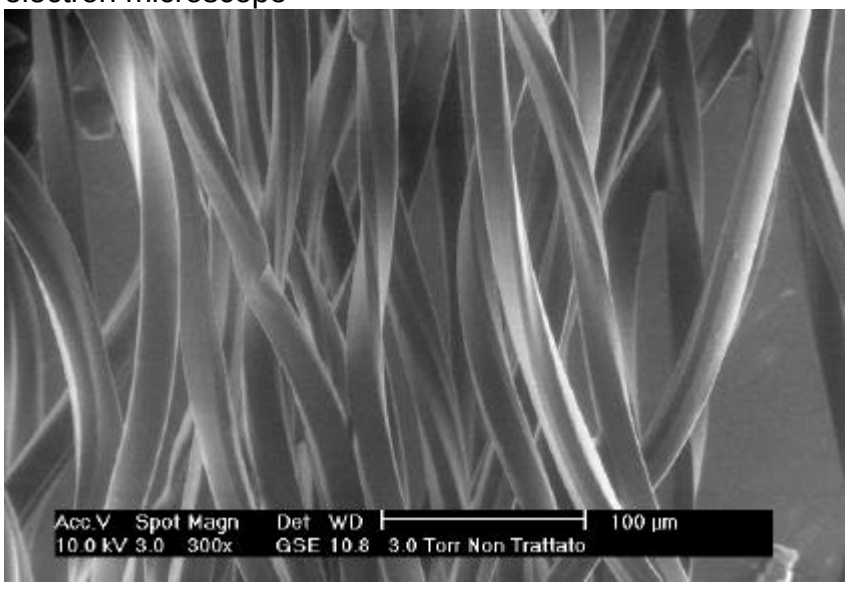


Reduced concentrations of oligomer are also seen after batchwise scouring with a similar recipe:

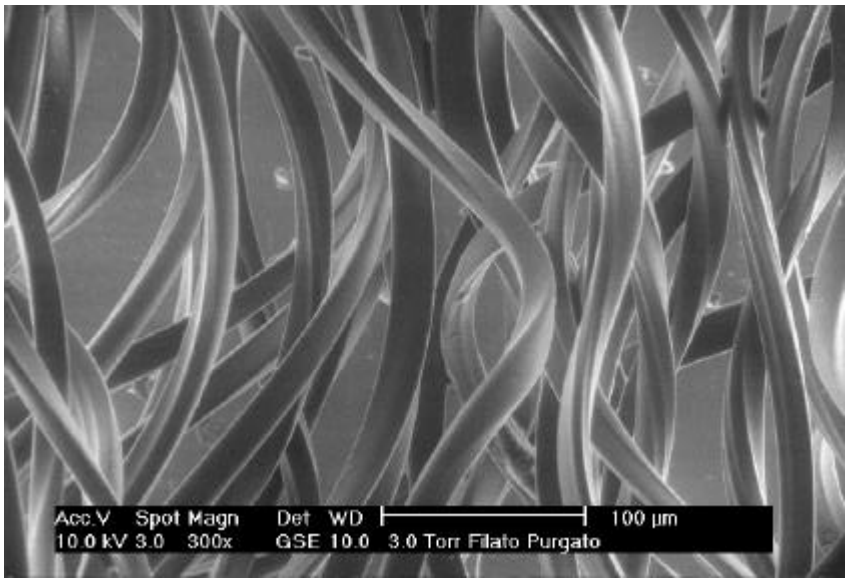
Nearpon TKR	1g/l
Nearchel TMC	1g/l
Naistat 1350	3g/l

90°C for 20 minutes.
rinse

Polyester yarn scoured with the above recipe examined under a scanning electron microscope



Untreated polyester yarn



After scouring with:
Nearpon TKR, Nearchel TMC and Naistat 1350

It can clearly be seen that oligomer has migrated to the surface of the yarn.

We have found that after dyeing at 130° C much more oligomer is brought to the surface.

High temperature dyeing with Neargal DN in the bath to disperse oligomers.

Cyclic tris(ethylene terephthalate) is insoluble in water. Solubility is increased in the presence of carriers, particularly of the methyl salicylate, alkyl phthalate and benzyl benzoate types, and such compounds, present in some levelling agents, like Neargal HP-MF NEW, promote migration of the trimer to the surface of the fibre. But a dispersing agent should be present to try to prevent crystallisation and deposition of the oligomers.

The longer the dyeing time the more oligomers will be released. Dark shades may produce more oligomer than paler shades because the dyeing time is longer.

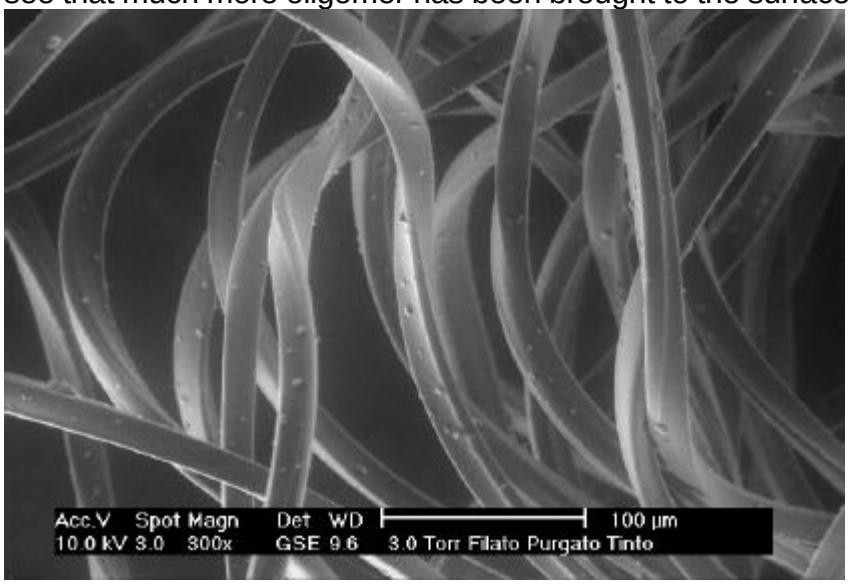
Neargal DN is a dispersing agent which is very stable to high temperatures. Many competitors' dispersing agents are NOT stable to high temperature. Thus, when oligomers are released from the polyester fibre, after some time at 130° C, there will not be an efficient dispersing agent in the bath to prevent crystallization of the oligomers, and to prevent small particles of oligomers coming together to form larger agglomerates, and depositing on the yarn and the machine.

Neargal DN does not magically **remove** the oligomers., but it will maintain the oligomers in a **fine dispersion** so that when the machine is drained more oligomer goes down the drain.

Discharging the dyebath at high temperature if this is possible can also reduce oligomer deposition.

We recommend the following recipe in the dyebath:
Dyeing
Neargal DN 0.5 to 2g/l (with serious oligomer problems – try 2 g/l)
Neargal HPMF New 1g/l
Nearchel TMC 1g/l
pH buffer
130°C for 30 minutes

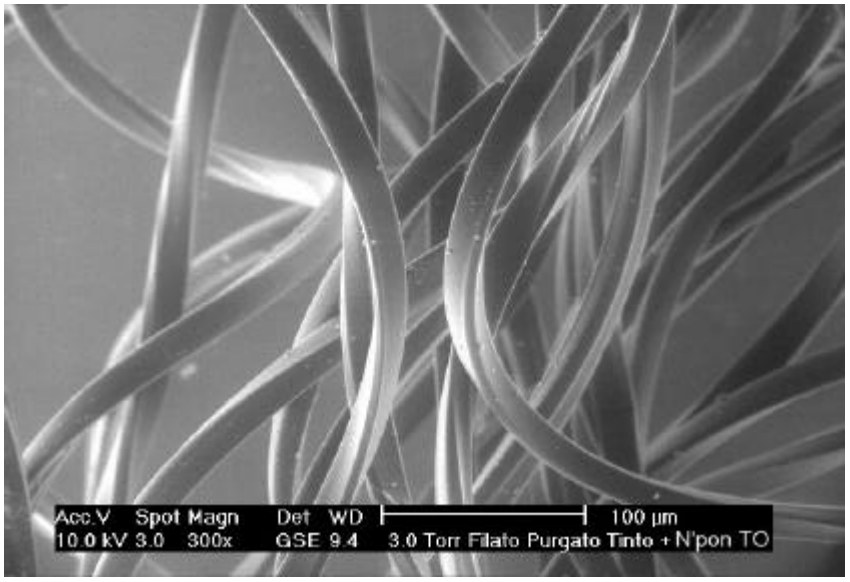
If we examine yarn under the scanning electron microscope after dyeing, we see that much more oligomer has been brought to the surface.



Yarn after scouring and dyeing.

Now we recommend that a further dispersing agent is used during reduction clearing of the polyester yarn.

Nearpon TO is a combination of non-ionic and anionic dispersing agents which helps to remove more oligomers during the reduction clear process. Sodium hydrosulphite reacts too quickly at 90° C, and the reducing power will be lost very quickly. Riducente FBT reacts more slowly at higher reduction clear temperatures, so that the clearing bath retains its reducing power for a longer time.
We recommend to reduction clear as follows:
Reduction clear
caustic soda 36° Bé liquid 5g/l
Riducente FBT 5g/l
Nearpon TO 2g/l
90°C per 15 - 20 minutes
2 rinses



Scanning electron microscope after scouring, dyeing, and reduction clearing with Nearpon TO.

Application of an oligomer-binding agent to polyester yarn in the last rinse on the dyeing machine can reduce the build-up of white powder deposits on the winding machine mechanism with polyester containing high concentrations of oligomer

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We have recently done trials with **Naistat 11009** and we see less build-up of white oligomer powder on the winding machinery.

Rinse in:
Naistat 11009 1 g/l

This may be combined with the softener, lubricant or anti-static agent normally applied.