## WHITE PAPER

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## AVOIDING GELS AND BLACK SPECKS IN POLYETHYLENE EXTRUSION

John Cowan (Senior Technologist) and Kim Fairhall (Product Integrity Manager) August 2015 Find out what causes gels and black specks and how they can be eliminated from the extrusion process

GELS AND BLACK SPECKS CAN OCCUR WHEN POLYETHYLENE RESIN BECOMES DEGRADED DURING THE PRIMARY POLYETHYLENE MANUFACTURING PROCESS OR THE PROCESS OF SECONDARY CONVERSION INTO A FINISHED PRODUCT. SUCH DEFECTS CAN SERIOUSLY COMPROMISE THE QUALITY, AESTHETICS AND PERFORMANCE OF ARTICLES MANUFACTURED FROM POLYETHYLENE RESIN.

Fortunately it is relatively easy to minimise the chances of gels and black specks occurring through the application of the correct quality assurance (QA) processes and procedures. This white paper explores the different types of gels and black specks that can form during the production and conversion of polyethylene and the best practices for avoiding their occurrence.

#### WHAT ARE GELS AND BLACK SPECKS?

Gels and black specks are visual or structural defects that can occur when polyethylene resin has become degraded. Such defects typically occur when polyethylene is exposed to high temperatures for a prolonged period while oxygen from air is present and are often observed within the first hour of a new production run.

Gels are usually formed at one of three stages during production:

- In the polymer reaction process itself as the raw polyethylene is manufactured
- In the granulation process when the product from the reactor is being mixed with additives and compounded into granules
- In the conversion process where the polyethylene is extruded and moulded into a finished article, for example during the process of blow moulding

There are two broad types of gels, with each type characterised by the manner in which it was formed. The first type is created during the reaction process and is caused by an accumulation of raw polyethylene particles growing within the reactor and producing material which has a significantly higher molecular weight than normal. These gels are colourless.

The second type is formed during the processing of the resin as it is run through equipment such as an extrusion or moulding line. This type of gel occurs due to a degradation of the resin which is typically caused by exposure to high temperatures for a long time in the presence of oxygen. Such gels tend to be cross-linked and are identifiable as they have a different structure to the gels produced in the reactor.

If a gel has remained at high temperature in the extruder for an extended period of time, it is possible for it to oxidise further and eventually discolour, creating brown or black specks within the finished product.

This type of gel often arises from hang-up of resin somewhere in the extrusion system. Such gels are more often observed during the first hour of a new production run, but can occur at any time if the equipment or conditions are not optimum.

Gels and black specks can arise when polyethylene resin becomes degraded at any point during the conversion process. This type of degradation typically occurs when polyethylene is exposed to high temperatures for a long time while oxygen is present. Once formed, such defects can seriously compromise the quality, aesthetics and performance of articles manufactured from polyethylene. The solution to avoiding such issues lies in the control of equipment operating temperatures, the amount of oxygen that is present and the shut down and start-up procedures.

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CONTROL OF OPERATING TEMPERATURES, THE PRESENCE OF OXYGEN AND THE START-UP AND SHUT DOWN PROCEDURES IS CRITICAL.

# WHICH APPLICATIONS ARE MOST SUSCEPTIBLE TO GELS AND BLACK SPECKS?

Gels and black specks are much more problematic in certain polyethylene applications than others.

Blow moulding is an application where gels and black specks can be more troubling, particularly in the production of thin walled bottles. In this application, the presence of gels and black specks can lead to the formation of holes and cause blow outs – a structural issue, rather than merely an aesthetic problem.

Even if the defect doesn't necessarily lead to holes, it can be visible on the surface and diminish the apparent quality of the article. This can be especially pronounced in products such as milk bottles where black specks will easily stand out.

Film extrusion is another application where gels and black specks can be problematic. Excessive defects in film affect the appearance of the film and can cause issues with printing. In applications such as the packaging of high quality goods, these defects can detract from the consumer's perception of the product quality. In the worst circumstances, severe gels which may occur particularly at the start-up or change in a job and lead to holes in the film causing significant production problems.

So what are the steps that can be taken to reduce the likelihood of them appearing?

### AVOIDING GELS AND BLACK SPECKS

Polyethylene producers typically use a range of controls to avoid producing resin with gels, including

- Precise control of reactor conditions
- Specific procedures for the maintenance of the reactor
- Close control of compounder conditions
- Strict procedures for shut down, start-up and purging the compounder
- Detailed procedures for the maintenance of the compounder
- Frequent and thorough quality testing of the product

Sourcing resin from a high quality supplier will reduce the risk of the presence of gels in the raw material.

Many polyethylene resins contain additives such as antioxidants which are added during the manufacturing process and serve to stabilise the resin and increase the resistance to degradation. Your resin supplier will advise you on the raw materials that are most appropriate for your process.

In the conversion process, polyethylene resin can adhere to the metal surfaces of the machinery in areas of low flow and degrade over time. When the equipment is shut down, the metal surfaces cool down and the degraded material can dislodge. When the equipment is restarted the degraded polyethylene resin may emerge and present with the appearance of black specks. It is essential to follow the correct machinery operation and maintenance guidelines in order to minimise the occurrence gels and blacks specks. The key is controlling the machine operating temperatures, the amount of oxygen that is present and the start-up and shut down procedures.

Close management of machine start-up and shutdown times is critical. The times can be optimised to minimise the time the resin is heated and exposed to oxygen as much as possible.

- The start-up of the various components of the equipment can be staged to minimise gel and black speck formation. For example, the head of a blow moulding machine usually takes the longest to heat up so this can be switched on first. Similarly, in film extrusion, it may be preferable to start heating the die earlier than the extruders.
- At shutdown, it is recommended to leave the extruder full of resin to prevent the entry of oxygen.
- It is also important to reduce temperatures as quickly as possible at shutdown. Setting the temperatures to a low level after extrusion is terminated and using the barrel cooling system to reduce temperatures is preferable to letting the machine sit and cool down naturally.
- Maintenance of equipment is also critical. Worn extruder screws and barrels can cause disrupted flow of the material and increase the residence time of resin in the extruder.
- The use of masterbatches that either contain antioxidants to minimise degradation or contain cleaning agents that help purge the machines and remove the defects may also be considered.

It should also be noted that reprocessed material may have depleted antioxidant levels and be more susceptible to degradation.

#### HOW THE RESIN SUPPLIER CAN HELP

Effective QA at each stage of the manufacturing process is critical in identifying issues and intervening before product quality is affected.

The Qenos Technical Service team has assisted many customers in overcoming issues related to the formation of gels and black specks on their processing equipment. Qenos has developed a state-of-the-art process for the optical detection, measurement and statistical analysis of the size, frequency and distribution of gels present in polyethylene resin. This is a cornerstone test in the quality assurance of Qenos produced resin.

At its Technical Centre in Melbourne, Qenos has an extensive range of processing equipment to allow the process simulation of all key segments in the Australian PE market. This local support, in conjunction with Qenos' highly experienced technical service specialists, is utilised to directly assist application development and troubleshooting for customers.



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