The Resistance of PEEK-OPTIMA® Polymer to High Temperature Autoclave Sterilisation

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1. Introduction

Steam sterilisation is commonly used in the clinical environment (e.g. hospital) as a means of sterilising medical products prior to use. The steam conditions and duration can vary depending upon the standards employed. In this study we selected 134°C steam for an extended period of exposure (1000hrs) in order to test the endurance of PEEK-OPTIMA under these conditions. Steam conditioned and control samples were assessed by mechanical testing, physiochemical analysis and biocompatibility testing.

2. Method

A number of injection moulded ISO test samples were prepared from Batch Number VPB1126 (PEEK-OPTIMA® LT1 polymer). Half of these were conditioned, being subjected to 134°C steam at 2 Bar pressure for 1000hrs at ICI (Measurement Science Group) Wilton UK. The remaining half were tested as control samples, having received no steam conditioning.

Both conditioned and control sample sets were subsequently assessed by way of standard Invibio® test procedures in order to ascertain any changes in mechanical performance, molecular weight or composition.

Mechanical:

Injection moulded ISO test bars were used to assess tensile strength at yield and elongation (ISO 527), flexural (3-point bend) strength and modulus (ISO 178) and impact strength (Notched Izod ISO 180). All tests were conducted under standard conditions at 23°C.

Thermal:

Differential Scanning Calorimetry (DSC) was used to measure the major transitions T_g, T_m T_c (glass, melt and re-crystallisation). Heat up rates were 20°C per minute in a sealed pan.

Molecular Weight:

Molecular weight was measured by Gel Permeation Chromatography (GPC) using polystyrene equivalent molecular weight calibration. The results are presented at weight average, number average molecular weights and polydispersity.

FTIR:

FTIR spectra were recorded using a surface reflectance method (golden gate) and pyrolysate method. The resulting spectra were compared with corresponding PEEK-OPTIMA LT1 reference spectra and the degree of coincidence reported.

Colour:

Colour was measured using a Minolta colour measuring system. Results are presented as L, a,b values (L= lightness, +a is positive red, +b is positive yellow)

In addition, samples of the steam conditioned material were subjected to cytotoxic analysis (ISO 10993-5) and chemical analysis (ISO 10993-18) in accordance with the ISO guidelines on assessing biomaterials (ISO 10993-1)

3. Results

The mechanical properties measured before and after steam conditioning are presented in Table 1. Also shown are the DSC results. The specification values are presented for reference.

Molecular weights are presented as weight average and number average molecular weights in Table 2. Table 3 shows the results from the FTIR analysis in terms of 'degree of coincidence'. Table 4 shows the results of the colour measurement tests.

Table 1.
Mechanical Properties and DSC

VPB 1126 PEEK-OPTIMA®LT1

Test	Test Method	Specification	Control	Conditioned (1000hrs Steam)
Tensile Strength	ISO 527	90MPa Min	99.58 MPa	107.69 MPa
Tensile Elongation	ISO 527	15% Min	37.06%	21.14 %
Flexural Strength	ISI 178	110 Mpa Min	159.27MPa	173.65MPa
Flexural Modulus	ISO 178	3.0 Mpa Min	3.98GPa	4.21GPa
Impact Strength (Notched Izod)	ISO 180	5.0 KJ/m² Min	7.0KJ/m²	7.1KJ/m²
Density	ASTM D792	1.28-1.30 g/cc	1.30 g/cc	1.30 g/cc
DSC	(20°C/min)			
Tg(onset)		135-155 °C	145.34°C	145.87°C
Melt Temperature		330-350 °C	342.95°C	342.29°C
Re-crystallisation Temperature		270-310 °C	290.02°C	289.51°C

Table 2. Polystyrene Equivalent Molecular Weight by GPC

Sample	Mw	Mn	Polydispersity
VPB 0001 (LT1Reference)	106,000	34,600	3.1
	105,000	34,700	3.0
VPB1126 After Steam	104,000	29,500	3.5
	104,000	29,700	3.5

Table 3. Search Results for Pyrolysate and Golden Gate IR Spectra

ACTUAL % FIT	LT1 VPB1126 After Steam
Pyrolysate IR	99.24%
Golden Gate IR	94.36%

A slight "Yellowing" effect was noticeable on the samples which were returned from steam resistance. These effects were quantified using the Minolta measuring system in the Victrex QC laboratory.

Table 4. Colour Measurement Results

Measurement	L	a	b
Control	58.68	+ 0.51	+11.61
Conditioned	61.95	+0.51	+15.05

Where L = Lightness
+a = positive red
+b = positive yellow

Biocompatibility

The material remained biocompatible following steam treatment.

4. Discussion

PEEK-OPTIMA® polymer displayed a remarkable degree of durability in the steam environment. All of the mechanical properties remain well within the specification for this grade. There was a moderate increase in the tensile strength, flexural strength and modulus values and a slight reduction in the strain to failure. These changes are speculated to be as a result of slight increases in crystallinity and/or reductions in internal stresses as a consequence of elevated temperature exposure close to the T_g (145°C). While some moderate yellowing was recorded, the FTIR spectra showed excellent correlation with the reference spectrum within normal expectations for PEEK-OPTIMA. The overlays of the reflection infrared spectra were very similar in terms of the fundamental infrared absorptions. An additional small broad absorption was detected in the region 3100-3500 cm^{-1} in the steam resistance sample. This absorption is consistent with the presence of small amounts of water still being present in the polymer matrix after the steam conditioning process. GPC analysis indicated a small change in the number average molecular weight and a subsequent increase in the polydispersity.

5. Conclusion

PEEK-OPTIMA polymer is very resistant to steam sterilisation for extensive periods (1000hrs) with only moderate changes occurring to the polymer. These results would be expected as a consequence of the ageing process for the PEEK-OPTIMA® material over this time period and temperature. Important is the fact that the material remains well within specification (mechanicals) and is demonstrated to be non-cytotoxic with regard to leachables as tested in accordance with ISO 10993-5.