High molecular weight HALS in LPDE

SpeedExtractor E-916:
Quantification of high molecular weight HALS in LPDE comparing Soxhlet extraction and pressurized solvent extraction using the SpeedExtractor E-916

The amount of high molecular weight hindered amine light stabilizer (HALS) in low density polyethylene (LDPE) was determined using two pretreatment methods; Soxhlet extraction and pressurized solvent extraction (PSE). The recoveries of high molecular weight HALS obtained by the two methods were compared. It became apparent that PSE is capable of obtaining much higher recovery rates of high molecular weight HALS in polymeric materials in a faster time than the Soxhlet extraction.

1. Introduction

High molecular weight HALS is widely added to polyethylene (PE) film or polypropylene (PP) film to enhance light and heat resistance. The quantification method of high molecular weight HALS is limited due to its low extraction efficiency [1]. Therefore, a method to easily extract high molecular weight HALS from polymers is desired. The PSE method is different from common extraction methods, such as Soxhlet extraction, as it uses solvent at high temperatures and pressures which improves extraction efficiency. The PSE method is easily performed using the SpeedExtractor E-916 made by BÜCHI Labortechnik AG. In this investigation, the amount of high molecular weight HALS in low density polyethylene (LDPE) was determined using two pretreatment methods; Soxhlet extraction and PSE. The recoveries of high molecular weight HALS obtained by the two methods were compared.

2. Experimental

Equipment: SpeedExtractor E-916 (BUCHI), GC/MS (Agilent Technologies), Soxhlet glassware

Samples: Polyethylene with high molecular weight HALS: ADK STAB LA62 (LA62) and ADK STAB LA63p (LA63), ADEKA corporation.

Fig 1: SpeedExtractor E-916

Determination: High molecular weight HALS 0.1 wt% were kneaded into LDPE using a high temperature roll. The test sample was cryogenically crushed to a fine powder. Pressurized solvent extraction was performed at 110 °C, 150 bar with 10 cycles of 10 minutes each. The overall extraction time was approx. 3 hours. The Soxhlet extractions were carried out for at least 16 hours. Methanol, dichloromethane and heptane were used as solvents. High molecular weight HALS were quantified by GC/MS.

3. Results

As shown in Table 1, recoveries of LA62 and LA63 were dependent on the type of solvents used. The recoveries with dichloromethane were higher than those with methanol. Furthermore, the Soxhlet extraction with dichloromethane was carried out for 48 hours. The recoveries of LA62 and LA63 only increased to approx. 50%. PSE using methanol yielded the highest recovery, as shown in Table 2, obtaining a recovery of 98% for LA62. High molecular weight HALS could not be extracted with dichloromethane or heptane by PSE.

Table 1: Recovery ratio [%] by Soxhlet extraction

Extraction solvent	Methanol	Dichloromethane	
Extraction time	16 h	16 h	48 h
LA62	44	49	51
LA63	16	31	48

Table 2: Recovery ratio [%] by PSE using the SpeedExtractor E-916

Extraction solvent	Methanol	Dichloromethane	Heptane
Extraction time	3 h	3 h	3 h
LA62	98	< 20	< 20
LA63	57	< 20	< 20

4. Conclusion

The PSE method using the SpeedExtractor E-916 is capable of obtaining higher recovery rates for high molecular weight HALS in polymeric materials in a faster time than the Soxhlet extraction.

5. Acknowledgement

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6. References

[1] L. Coulier, E. R. Kaal, M. Tienstra, T. Hankemeier; J. Chromatogr. A, 1062, 227-238, (2005)

· Operation Manual SpeedExtractor E-916/E-914

For more detailed information please refer to the Application Note no. 151/2014.