Pomegranate extract for the processing stabilization of polyethylene

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Abstract

Active components with antioxidant activity were extracted from the powder of pomegranate peel and added to polyethylene at 1000 ppm. The stabilization effect of the extract was studied also in compounds containing a phosphorous secondary antioxidant. A commercial hindered phenolic antioxidant with and without a secondary stabilizer was used as reference. The polymer was processed in multiple extrusions, and its functional group content, processability, residual stability, and color were determined by Fourier-transform infrared (FTIR) spectroscopy, melt flow rate (MFR), and oxidation induction time (OIT) measurements as well as by the determination of the yellowness index. The pomegranate peel extract (POM) has very strong melt stabilization effect, which is comparable or better than that of the commercial hindered phenolic antioxidant used as reference. The extract hinders the reactions of the unsaturated groups of the polymer and prevents the formation of long-chain branches. Similar to the phenolic antioxidant, it hinders also oxidation, thus preventing the formation of carbonyl groups. On the other hand, the extract does not offer sufficient residual stability and the polymer stabilized with it cannot be used in long-term applications. The extract discolors the polymer somewhat that further limits its application possibilities. The contradiction of excellent processing stabilization and poor residual stability needs further study and explanation.

KEYWORDS

antioxidants, FTIR, polyethylene (PE), processing, stabilization

1 | INTRODUCTION

Polyolefins, including polyethylene (PE), are commodity polymers that are used in very large quantities in all areas of life. Thermoplastic polymers are processed at relatively high temperatures by various melt technologies. The polymers are subjected to heat, shear, and oxygen during these processing operations. PE undergoes thermo-oxidative degradation through radical chain reactions as an effect\cite{1}. In order to protect the polymer, PE is stabilized routinely by the combination of a phenolic antioxidant and a secondary stabilizer, usually a phosphate or phosphonite\cite{2,3}. Light stabilizers might be also added to the additive package, in applications that require it. The current stabilization
practice is well established; thus very few new stabilizers appeared on the market recently, and the development is limited to combined additives offering synergistic effects\(^5\) or technological advantages (non-dust blends).\(^6\) However, some years ago, concerns were raised about the possible environmental and health hazard of the reaction products of phenolic antioxidants,\(^7\) and most of the related questions have not been answered yet. Natural antioxidants are already extensively used in the food industry,\(^8\) and they might offer a new way of stabilization also for polymers.

Living organisms, including plants, produce and use a large number of antioxidants very efficiently. These compounds can have diverse structures, functions, and efficiencies. Quite a few of them have been tried as stabilizers also in polymers with different successes. Carotene,\(^9\)–\(^11\) curcumin,\(^12\) vitamin E,\(^13\)–\(^15\) lignin,\(^16\)–\(^18\) and many other compounds have been shown to have smaller or larger stabilizing activity in a range of polymers, but mostly in PE. Recently, the interest in natural antioxidants has increased considerably and a number of papers have been published on them.\(^19\) According to these articles, flavonoids seem to be the most efficient natural stabilizers at least in PE. Quercetin,\(^20\) dihydromyricetin,\(^21\) and rutin\(^22\) provided sufficient melt stability for PE already at 50 ppm and residual oxidative stability at 250 ppm. All three are considerably more efficient than the phenolic antioxidant used in the largest quantity in current industrial practice. Although natural antioxidants are very efficient, they have several drawbacks as well, their melting temperature can be very high, higher than the processing temperature of PE, their solubility is small, and they discolor the polymer.\(^19\)

Although the extraction of active components from plants is an industrial practice now, obtaining a pure compound is still tedious partly because of the large number of components and partly because of the small amount of a single compound. The direct use of a natural extract for stabilization would be more advantageous, and a recent study showed that the natural extract of silymarin was a more efficient stabilizer than the main component of the extract, silybin.\(^23\) Pomegranate (Punica granatum L.) is cultivated throughout the world in subtropical and tropical areas, mainly in Mediterranean and Asian countries and in several countries of North and South America.\(^24\) Pomegranate is produced in large quantities in India, China, Iran, and Turkey; India is the main exporter of pomegranate in the world.\(^25\) The fruit has been used extensively as traditional medicine in many cultures.\(^26\)–\(^27\) Several studies indicated that the extract of pomegranate peel had much stronger antioxidant activity than the seeds.\(^28\)–\(^31\) Because of the huge amounts of by-products forming in the industrial processing of the pomegranate fruit,\(^25\) recent studies have focused on the antioxidant activity of seeds and peels. Pomegranate peel extract (POM), for example, was applied in the food industry; it increases the shelf life of natural oils.\(^32\)–\(^33\) The incorporation of POM into poly (vinyl alcohol)\(^34\) fish gelatin,\(^35\) chitosan,\(^36\)–\(^37\) and chitosan–polyvinyl alcohol\(^38\) matrices increased the antioxidant and antibacterial activities of the packaging material. Although the antioxidant activity of pomegranate extracts has been proved several times, according to our knowledge, only a single attempt has been made to try to use it as an antioxidant in polyolefins,\(^39\) and several questions remained unanswered even in this case.

In accordance with the increasing tendency of using materials from renewable resources and considering the results obtained on the antioxidant activity of pomegranate extracts, the goal of this study was to extract active components from pomegranate peel and use the extract as stabilizer in PE. As indicated in the previous paragraph, natural antioxidants have a beneficial effect on food packaging; thus, the targeted area of application of our product can be the manufacturing of products with short service life like films, boxes, and caps. Processing stabilization and residual stability were determined in order to see the possible application of the extract in industrial practice. The hindered phenolic antioxidant used in the largest quantity in industry, Irganox 1010, was used as a reference stabilizer. The antioxidants were applied in packages containing a phosphonite secondary stabilizer as well. Besides processing stability, structural changes in the polymer as well as other aspects, like residual stability and color, were also considered. The possible practical consequences of the results are also discussed briefly in the final section of the paper.

### 2 | EXPERIMENTAL

#### 2.1 | Materials

The polymer used in the experiments was the Tipelin FS 471 grade ethylene/1-hexene copolymer (melt flow rate [MFR]: 0.3 g/10 min at 190°C, 2.16 kg; nominal density: 0.947 g/cm\(^3\)) polymerized with a Phillips catalyst. The additive-free polymer powder was provided by the MOL Group Ltd., Hungary. The POM was prepared from dried pomegranate peel powder, obtained from Turkey, by Soxhlet extraction using acetone as solvent at its boiling point (56°C). The extraction was carried out until the complete depletion of the powder of active components, approximately 7 h daily for 4–5 days. Acetone was removed by vacuum distillation resulting in a yellow, lightly scented powder. The POM was added to the polymer at 1000 ppm. Since industrial packages also contain a secondary antioxidant practically always, the combination of 1000 ppm POM and 1000 ppm Sandostab PEPQ.
(PEPQ, Clariant, Switzerland) was also used for stabilization. The polymer was stabilized also by 1000 ppm Irganol 1010 (1010I, BASF, Germany) alone and in combination with 1000 ppm PEPQ for comparative purposes. The polymer containing PEPQ alone and the neat PE powder were also studied as references.

2.2 Sample preparation

The additives were mixed with the PE powder and then homogenized in a high-speed mixer (Henschel FM/A10, Germany) at the rate of 1000 rpm for 10 min. The POM was added to the powder in the form of acetone solution to achieve the best homogeneity possible. The powder was air-dried overnight and extruded on the next day. The dry blend was processed and pelleted in six consecutive extrusion steps at 50 rpm and the barrel temperatures of 180, 220, 260, and 260°C using a Haake Rheomex 1/2” (USA) type single-screw extruder attached to a Haake Rheocord EU 10 V driving unit (USA). Samples were taken after each extrusion step. For further studies, films of about 100 μm thickness were compression molded at 190°C and 5 min (3 min preheating and 2 min compression) using a Fontijne SRA 100 (The Netherlands) machine.

2.3 Characterization

The MFR of the polymer was determined according to the ASTM D 1238–79 standard at 190°C with 2.16 kg load using a Göttfert MPS-D (Germany) MFR tester. Five parallel measurements were done on each sample. Residual thermo-oxidative stability was characterized by the oxidation induction time (OIT) measured at 190°C in oxygen atmosphere at a constant 20 ml/min flow rate in open aluminum pans using a Perkin Elmer DSC-7 (USA) apparatus. The measurements were carried out principally according to the ISO 11357-6:2018 standard, but the temperature was changed to 190°C because of the limited residual stability of the materials. OIT was determined in triplicates. The concentration of the unsaturated functional groups of PE was determined by Fourier-transform infrared (FTIR) spectroscopy on the 100–μm-thick compression-molded films in transmission mode using a Bruker Tensor 27 (Germany) spectrophotometer. Five parallel measurements were carried out on each sample between 4000 and 400 cm⁻¹ wavelengths at 2 cm⁻¹ resolution by 16 scans. The concentration of vinyl,[40,41] carbonyl,[42,43] and vinylidene[44] groups was determined from absorbance spectra by the internal standard method according to Equation (1):

\[
C = \frac{K}{\rho} \left( \frac{A_{\text{inv}}}{A_{\text{ref}}} \right),
\]

where \(C\) is the concentration of a given functional group (group/1000 C atom), \(K\) is its absorption coefficient, \(\rho\) is the density of the polymer (g/cm³), while \(A_{\text{inv}}\) and \(A_{\text{ref}}\) are the intensities of the investigated and the reference absorption bands, respectively. We used the absorption band of the –CH vibration appearing at 2018 cm⁻¹ as reference. A series of Phillips-type ethylene/1-hexene copolymers with various butyl branching and unsaturations was investigated earlier to determine the absorption coefficient of vinyl group vibration (band at 908 cm⁻¹; \(K = 0.5000\)). The vinylidene concentration was determined from the absorption band at 888 cm⁻¹ \((K = 0.5654)\).[44] The concentration of the carbonyl groups was calculated from the integrated absorption intensity of the vibration appearing in the range of 1780–1690 cm⁻¹ \((K = 0.03826)\).[42,43] FTIR spectroscopy was used also for the determination of residual PEPQ content (ppm) based on the absorption of the P(III)-O-C groups at 850 cm⁻¹ \((K = 4955)\).[45] The color of the samples was described by the yellowness index (YI) determined by using a HunterLab ColorQuest 45/0 (USA) apparatus.

3 RESULTS AND DISCUSSION

The results are discussed in several sections. The composition and antioxidant activity of the POM are considered in the first section, and then, its effect on the properties of the polymer in comparison with the industrial package is presented next. The influence of the stabilizers on residual stability and color is shown in the following section, while correlations as well as consequences for practice are discussed in the last section of the paper.

3.1 Composition and antioxidant effect

As mentioned in the introductory part, pomegranate (Figure 1) has been used in the traditional medicine of many cultures for a long time.[46] It offers remedy to a great number of diseases and ailments including high blood pressure, high cholesterol, oxidative stress, hyperglycemia, and inflammatory activities. It was demonstrated that pomegranate has potential antioxidant, anti-inflammatory, and anticarcinogenic effects.[47] Studies have shown that pomegranate juice and even fermented pomegranate juice possess large antioxidant activity,[48,49] which is related to various phenolic
The pomegranate fruit and its components

Because of its beneficial effect on health, the interest in pomegranate is large and a considerable number of studies have been carried out to analyze its composition and identify its active components. Ambigaipalan et al., for example, found that pomegranate peel contained 79 phenolic compounds, including 16 phenolic acids, 12 flavonoids, 35 hydrolyzable tannins, 8 proanthocyanidins, and 8 anthocyanins. Phenolic acids were the major phenolic compounds in pomegranate peel followed by hydrolyzable tannins, proanthocyanidins, and flavonoids, mostly present in the insoluble-bound form. Punicalagin with high molecular weight is the most abundant component in pomegranate peel, and it is considered as a characteristic compound of this part of the fruit. Rongai et al. found the concentration of punicalin in dried pomegranate peel being between 1.6–476 mg/g depending on the genotype. Fischer et al. analyzed extracts and juices of pomegranate by high-performance liquid chromatography with a diode-array detector–electrospray ionization multistage mass spectrometry and found 48 compounds in them in total. They identified 9 anthocyanins, 2 gallotannins, 22 ellagitannins, 2 gallagly esters, 4 hydroxybenzoic acids, 7 hydroxycinnamic acids, and 1 dihydroflavonol in the fruit. Somewhat surprisingly, they found only 0.13 mg/g of punicalin, which is another main component in pomegranate, but Li et al. detected around 10 times larger amount of this component. These examples clearly show that pomegranate extracts contain many components, and their complete identification and quantitative determination are practically impossible. Components and quantities depend on location, source, extraction technique, etc.

Similar to components and composition, also the information on the antioxidant activity of the fruit, or more exactly the extracts, varies in a wide range. The antioxidant activity of pomegranate extract is estimated in various ways mostly by the reaction with the stable free radical, 2,2-diphenyl-1-picryl-hydrazyl (DPPH). The DPPH assay expresses activity by the effective concentration (EC_{50}) or inhibition concentration (IC_{50}) value, which is defined as the concentration of the substrate that results in a 50% loss in DPPH activity. The EC_{50} value for punicalagin was found to be 54.2 μg/ml, while the methanol extract of pomegranate had an EC_{50} value of 32.4 μg/ml showing larger activity than the pure compound. This may be due to the synergistic effect of other phenolic components present in the crude extract supplementing its radical scavenging ability. Although the results of the studies differ in components and composition, they all agree that activity is high but also that it depends on a number of factors. In spite of all the uncertainties, we may conclude from these studies that the extract of pomegranate peel has large antioxidant activity, and it may act as an efficient antioxidant also in PE.

### 3.2 Processing stabilization

Numerous reactions take place during the processing of polymers at the relatively high temperature of the operation. Shear and the small amount of oxygen present increase the probability of degradation even further. The reactions taking place result in the modification of the structure of the polymer, that is, molecular weight can either decrease or increase as the result, and various functional groups (unsaturations, carbonyls, etc.) may also form on the polymer chains. The Phillips polymer used in this study was shown to form long chain branches during processing mostly through the addition of C-centered alkyl radicals to its chain-end vinyl groups. The changes in the vinyl group content of the polymer during multiple extrusion are presented in Figure 2 for the various additive packages compared. As expected, a large number of vinyl groups are consumed during the processing of the neat polymer. The secondary antioxidant, the phosphonite used, hinders the reaction of the vinyl groups considerably, but not completely. The phenolic antioxidants are quite efficient in preventing the consumption of the vinyl groups; there is hardly any change in vinyl group content during the multiple extrusions. It is somewhat surprising that the combination of the commercial phenolic antioxidant, I1010, and the phosphonite seems to be slightly less efficient than the phenol alone or the POM and its combination with PEPEQ. Nevertheless, we may expect efficient melt stabilization from the experimental POM prepared in this study.

Although the main reaction of degradation is the addition of radicals to the vinyl groups for the polymer
used, other unsaturations, vinylidene, and trans-vinylene groups may also take part in reactions. Figure 3 presents the variation of vinylidene groups during the multiple extrusions. The original value in the virgin PE powder was 0.054 vinylidene group/1000 C atom. According to the results presented in Figure 3, vinylidene content increases during processing, but to very different extents in the presence of the various stabilizer combinations. Vinylidene groups form mainly by the reaction of tertiary radicals\(^{60-63}\) with intramolecular decomposition of special secondary hydroperoxide groups.\(^{64}\) The polymer used in this study is a 1-hexene copolymer; thus, it contains a certain number of tertiary carbon atoms. The phosphonite seems to facilitate the formation of this functionality, that is, vinylidene group; its amount is the largest when PEPQ is used alone, and some increase can be observed in all cases when the polymer contains it. However, we must keep in mind during evaluation that the absorbance band of oxidized PEPQ (890 cm\(^{-1}\)) overlaps with the absorption of the vinylidene group (888 cm\(^{-1}\)) apparently increasing this later. We must call the attention here to the fact, though, that the smallest number of vinylidene groups is detected in the polymer which is stabilized with the POM, indicating that it does not hinder only the reactions of the vinyl groups but also the formation of vinylidenes.

Polymers always contain some adsorbed or dissolved oxygen\(^{65}\); thus, oxidation usually takes place even under the oxygen-poor conditions of processing. Oxidation is frequently followed by the measurement of the total carbonyl content of the polymer. This quantity is plotted against the number of extrusions in Figure 4 for the compounds studied. The neat polymer not containing any antioxidant oxidizes indeed, and the number of oxygen-containing groups increases rather steeply in each extrusion step. PEPQ in itself prevents oxidation successfully, but a certain amount of carbonyls is detected in the rest of the compounds. However, the number of such groups does not increase significantly during multiple extrusions.
and we must consider that both I1010 and the POM inherently contain carbonyl groups, which are detected by FTIR spectroscopy. Accordingly, all the stabilizers as well as their combinations are efficient in preventing oxidation reactions during processing.

The simplest and fastest method for the determination of melt stability is the measurement of MFR, which sensitively detects any changes in the molecular structure of PE. The effect of multiple extrusions on the MFR of PE containing the various types and combinations of stabilizers is presented in Figure 5. The MFR of the neat polymer decreases drastically and reaches a very small value already after the third extrusion because long chain branches are forming during processing. PEPQ in itself slows down the increase of melt viscosity but does not prevent it completely. Both additives, the hindered phenol antioxidant, and the POM stabilize the polymer efficiently, and MFR does not change during multiple extrusions. A small decrease is observed after the first extrusion which is completely eliminated by the addition of PEPQ. The beneficial effect of phosphorous secondary antioxidants, and especially that of PEPQ, has been reported earlier.\textsuperscript{3,4} We may conclude, though, that the natural extract prepared in this study is at least as efficient melt stabilizer as the commercial hindered phenolic antioxidant.

Because of its beneficial effect, the consumption of the secondary phosphorus antioxidant was followed during the multiple extrusion of the polymer and it is shown in Figure 6. Obviously, only the compositions containing PEPQ are presented in the figure. The secondary antioxidant is consumed very rapidly when it is used as a single stabilizer, but its consumption is much slower in the presence of primary antioxidants. I1010 protects efficiently the secondary antioxidant in the first few processing steps, but PEPQ content goes to a very low level at larger number of extrusions. On the other hand, in spite of a large drop in the first extrusion step, the natural extract offers a prolonged protection for the phosphonite, which is especially beneficial in the case of high temperature and shear loads or when PE waste is reground and reprocessed several times.

### 3.3 Residual stability and color

Although the POM offered excellent melt stabilization for the PE used in this study, there are other aspects that must be considered during the evaluation of the efficiency of a stabilizer. Some plastic products have a short lifetime, but others are guaranteed for a long time, and thus, they must have long-term stability. This latter depends mainly on the amount of residual active phenolic antioxidant, although secondary antioxidants and other constituents of the stabilizer package may also influence its value. Residual stability is often characterized by the OIT. The residual stability of the PE compounds studied is presented in Figure 7 as a function of the number of extrusions. Irganox 1010 alone renders the polymer sufficiently stable, and OIT is around 60 min in its presence and does not change much with increasing processing history. The addition of the secondary antioxidant increases OIT even further, as expected, the synergistic effect, and mutual protection of the two stabilizers.
result in very good long-term stability. The neat polymer has practically zero stability, while the secondary antioxidant alone is not able to protect the polymer in the oxygen-rich environment of the OIT measurement. Quite surprisingly, the POM in itself, but also in the presence of PEPQ, does not offer sufficient long-term stability. The OIT of the polymer is around 10 min, which is not sufficient for long-term applications. Although the POM proved to be a very efficient processing stabilizer, it definitely cannot be used in applications in which longer lifetime is required.

Another important issue is the color of the product and the effect of the additives on it. Even hindered phenolic antioxidants discolor the polymer slightly, but the use of a phosphorus antioxidant sufficiently compensates for the effect. Natural antioxidants are either colored themselves\textsuperscript{[12,20,22,23]} or their reaction products give the polymer a strong color.\textsuperscript{[21]} The extract, as mentioned in the experimental part, has a slight yellowish color; thus, we might have expected only weak color in the product. The YI of the PE compounds is plotted against the number of extrusions in Figure 8. The neat polymer and the one containing only PEPQ do not have practically any color at all. I1010 discolors the polymer somewhat, but the color is taken away by PEPQ, as mentioned above. The natural extract gives a very strong color to the polymer, and the YI increases even further upon the addition of PEPQ that is rather surprising. Accordingly, we must conclude here that the POM cannot be used for the production of colorless or lightly colored products.

\section*{3.4 Correlations and discussion}

The processing stability of our Phillips-type PE depends very much on the reactions of unsaturated groups and especially that of the vinyl group. The comparison of Figures 2 and 5 clearly indicates that strong changes in vinyl group content are accompanied by the modification of MFR. The correlation of the two variables, that is, MFR and vinyl content, are plotted against each other in Figure 9. The figure confirms the statement above, that is, the number of vinyl groups and its change strongly influence the viscosity of the polymer. It is clear from the figure that below a certain level of vinyl content, at around 0.7–0.8 vinyl group/1000 C atom, MFR becomes very small and the processability of the polymer deteriorates very much. A steep increase of MFR can be observed above this level with increasing vinyl content. The efficiency of the various additives and their combinations varies, that is, the most efficient seems to be the I1010/PEPQ and the POM/PEPQ combinations.

Another related and important issue is the interaction of the primary and secondary stabilizers, that is, their ability to protect each other. The presence of the secondary antioxidant is crucial in many respects. Figure 10 shows the correlation of MFR and the amount of residual PEPQ, that is, the amount of phosphorous with three valence electrons. According to the figure, the total consumption of the phosphonite results in a rapid and drastic decrease of MFR, but obviously, the primary antioxidant also plays a role in the determination of viscosity.
One important result that has a strong relevance for practice is the contradiction observed in the case of the POM extract. The extract has a very strong melt stabilization effect that is comparable to that of the commercial hindered phenolic antioxidant. On the other hand, it does not offer any protection under the oxygen-rich conditions of the OIT measurement. The contradiction is very difficult to explain and needs further study and considerations. Nevertheless, it is obvious that this particular natural extract cannot be used for the stabilization of PE or any other polymer that is used in applications requiring long lifetime. A further disadvantage of the extract is that it discolors the polymer that also limits its possible application, although many products are dark or even black, especially in the automotive industry. The excellent processing stabilization efficiency of the extract deserves further study, but the contradiction mentioned above must be resolved before the extensive use of the material as a stabilizer in the polymer industry.

4 CONCLUSIONS

According to literature references, extracts prepared from pomegranate have numerous components with antioxidant activity. The composition of the extract depends on many factors, and the identification as well as separation of the components is difficult. The extract prepared in this study from the powder of pomegranate peel has very strong melt stabilization effect, which is comparable or better than that of the commercial hindered phenolic antioxidant used as reference. The extract hinders the reactions of the unsaturated groups of the polymer and prevents the formation of long-chain branches. Similar to the phenolic antioxidants, it hinders also oxidation, thus preventing the formation of carbonyl groups. On the other hand, POM does not offer sufficient residual stability, and the polymer stabilized with it cannot be used for long-term applications. The extract discolors the polymer strongly which further limits its application possibilities. The contradiction of excellent processing stabilization and poor residual stability needs further study and explanation. The use of natural extracts as stabilizers in polymers is a promising way to substitute synthetic phenolic antioxidants, but further research is needed for the clear identification of their advantages and limitations.

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