Phosphated Avocado Seed: A Renewable Biomaterial for Preparing a Flame Retardant Biofiller

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Abstract

The cellulose and starch present in the avocado seed can be chemically modified to obtain biofillers with fire retarding characteristics. The resulting composites could be used as substitute of the corresponding halogenated composites. For this, the avocado seed was first washed, dehydrated and pulverized, and thereafter, chemically modified with phosphoric acid in the presence of urea. This was studied using infrared spectroscopy, nuclear magnetic resonance and X-Ray photoelectron spectroscopy, in order to determine the resulting chemical structure and confirm the presence of the proposed functional groups. In addition, scanning electron microscopy and elemental analysis were used, respectively, to establish the resulting morphological changes, as well as the elements present on the surface of the modified material. Thermogravimetric analysis was also carried out in order to establish the thermal stability of the material and predict the effect on the flame retardancy due to the mentioned chemical modification. Further tests established that the obtained modified structure and morphology of the avocado seed was highly dependent on the method used to dehydrate the pulverized avocado seed. It was also determined that chemical modification greatly increased the thermal stability of the avocado seed in air atmosphere. The flame-retardant effect of the modified avocado seed was assessed in polyethylene/ethylene-vinyl-acetate (PE/EVA) composites via cone calorimeter tests. These results showed that the modified avocado seed decreased the peak of the heat release rate (pHRR) by 50% and the total heat released (THR) by 15%. This phosphated avocado seed could be a good option as a renewable biofiller for polymer composites with enhanced flame-retardant properties.

Introduction

Avocado, (*Persea Americana*, Mill) is a native fruit of Mexico and Central America. The total world production in 2014 was greater than 5 million tons, 30% of which were produced in Mexico, which is the greatest producer (FOASTAT 2014). In 2015, Mexico produced 1.6 million tons, and most of this is centered in the Hass (Araújo et al. 2018) variety. Avocado is worldwide commercialized as a fruit, and among its derivatives is the avocado oil and the processed pulp. The discarded parts of the avocado are the skin and the seed, which amount to ca. 25 wt% of the fruit (López-Cobo et al, 2016). Usually, these are not used and are discarded; however, these represent ca. 25 wt% of the 1.6 million tons produced per year, and have become a serious environmental problem (Padilla-Camberos et al, 2013). The main components of the residues during fabrication of avocado oil and processed pulp, as well as of the avocado skin and avocado seed are the carbohydrates, such as fibers, cellulose, hemicellulose and starch (in the seed) (Ruiz et al. 2013). The pulverized avocado seed can be incorporated as filler, into many polymeric matrices to reduce costs. Nevertheless, one of the main problems of adding biofillers, is that usually, these tend to thermally degrade, due to their low thermal resistance (Poletto et al. 2010), coupled to the high processing temperatures of most thermoplastics. As mentioned above, the avocado seed is mainly composed of cellulose and starch (Weatherby et al. 1931), both being polysaccharides composed of glucose units, where the structural difference is that in cellulose, the bonds between the glucose units are syndiotactic, whereas in starch the bonds are isotactic (Schmidt et al. 2013, Takada et al. 2015). The

avocado seed is abundant in cellulose and starch, whose chemical structures contain large amounts of hydroxyl groups. These groups allow covalent bonds between phosphoric acid and cellulose or starch, to form Phosphate-Cellulose-O-P(O)(OH)₃ (Hendrix et al. 1872) and Phosphate-Starch-O-P(O)(OH)₃ (Chowdary et al. 2011) groups. Terminal hydroxyl groups from the phosphoric acid covalently bonded to cellulose or starch, can form salts when in the presence of compounds with amino (NH₂) groups (Xiong et al. 2018). One such compound may be urea whose amino groups would react with the phosphate group in the cellulose or in the phosphated starch (Hassan et al. 2016). The flame-retardant effect of these phosphated materials is attributed to the dehydration of the hydrocarbon chain, which occurs due to the liberation of an acid source usually HPO₃, meta-phosphoric acid, which in turn promotes the formation of a char layer once the fire starts. This char layer is highly fire resistance and is known as intumescence. In addition, this char prevents the dripping of already ignited material (Pan et al. 2014). Previous studies report that the phosphating of cellulosic materials prevents the loss of mass during pyrolysis under oxidative atmosphere, that is, it imparts flame retardancy. Moreover, it has been reported that the addition of phosphated compounds into nitrogenated materials has a synergistic effect, providing a marked increase in flame retardancy (Leu et al. 2003, Gaan et al. 2008). However, nothing is mentioned if the material contains another substituent, apart from nitrogen, nor if these materials can be prepared from agricultural waste. In the present work it is proposed to use the pulverized, dehydrated, and modified avocado seed as a flame retarding filler. This was studied using infrared spectroscopy and nuclear magnetic resonance, in order to determine the resulting chemical structure and confirm the presence of the proposed functional groups. Finally, its flame-retardant effect on a polymer composite was assessed using the cone calorimeter technique.

Experimental Section

Materials and chemicals. Avocado seeds “persea americana” of the Hass variety, obtained from food waste of local restaurants, phosphoric acid 85% (H₃PO₄) from Fermont (México), urea 99.5% and sodium hydroxide (NaOH) from Sigma Aldrich. The Polymers used were low density polyethylene (LDPE) PE-20020X from PEMEX with a melt index of 2.2 g/10 min. Ethylene-vinyl acetate copolymer (EVA) ELVAX 360 was from DOW with 25% vinyl acetate and a melt index of 2.0 g/10 min.

Methods for Cleaning, Drying, and Pulverization of the Avocado Seed. The avocado seeds were first washed with water, then lyophilized until 4 wt% water content, and pulverized. The pulverization was carried out in two steps; first, the avocado seeds were milled in a Brabender TYP51079 blades mill grinder and second, the grinded seeds were dry processed in a Werner & Pfleiderer ZSK 30 twin screw extruder at 50 rpm and at 25°C. The milled material was passed through separation screens, and the material selected was the one that passed through a 325 mesh, which corresponds to 44µm. The material that passed the 325 mesh is the one referred as pulverized avocado seed along this manuscript.

Preparation of the Phosphated Avocado Seed. The phosphated avocado seed was prepared as follows: 12.5 g of urea were first dissolved in 25 ml of distilled water at 60°C, then, 2 g of the pulverized avocado seed plus 6 ml of phosphoric acid were slowly added and left for 3 h. The obtained, pulverized and

phosphated avocado seed was then washed 5 times with a Na (OH) 1N solution. In an alternate manner, another set of pulverized and phosphated avocado seed was obtained but washed with deionized water (H₂O).

Preparation of the PE/EVA-Avocado Seed Biocomposites. In order to determine the flame retarding characteristics of the pulverized avocado seed, PE/EVA (80/20wt%) biocomposites were prepared with 30 phr of the pulverized, unmodified as well as modified, avocado seed, using a Brabender internal mixer, working at 60 rpm and 190°C for 10 min. **Previous** to the addition of the pulverized avocado seed, the PE/EVA was processed for 5 min at 60 rpm and at 190°C, for melting of the PE/EVA part.

Characterization. *Attenuated Total Reflectance Infrared Spectroscopy (ATR-FTIR):* These analyzes were carried out in a Bruker VERTEX 70V infrared spectroscopy instrument, equipped with an ATR accessory. *Nuclear Magnetic Resonance ¹³C and ³¹P (NMR):* These tests were carried out in a Bruker Ascend NMR 400 MHz instrument at 25°C. All samples were dissolved in D₂O at a ratio of 10 mg/cm³. The ³¹P spectra were obtained using a conventional pulse with 100 scans, whereas the ¹³C spectra were obtained using a conventional pulse and an acquisition time of 12h. *Thermogravimetric Analysis (TGA):* These tests were carried out in a TGA – TA Instruments Q500 at a heating rate of 10°C/min, from 30 to 600°C, in a nitrogen atmosphere. *Scanning Electron Microscopy (SEM):* Qualitative chemical analysis of samples was carried out in a FEI Quanta 200-3D, coupled with an EDAX detector. Operating conditions were 15kV with a working distance of 15mm. All samples were previously coated with Au-Pd. *X Rays Photoelectron Spectroscopy (XPS):* The XPS spectra were collected with a Kratos Axis Ultra DLD electron spectrometer using a monochromatic Al-K α X-ray source operated at 150 W. An analyzer pass energy of 160 eV for acquiring wide spectra and a pass energy of 20 eV for individual photoelectron lines were used. Fiber samples for the analysis were gently hand-pressed using a clean Ni spatula into a special powder sample holder. The surface potential was stabilized by the charge neutralization system of the spectrometer. The binding energy (BE) scale was referenced to the C 1s line of aliphatic carbon, set at 285.0 eV. The spectra were analyzed with the Kratos software. *Cone Calorimeter:* Samples of 100x100x2 mm were tested in a Fire Testing Technology Cone Calorimeter, using a heat flux of 35 kW/m² and a separation of 25 mm between the cone and the sample, in accordance to ASTM E1354-14.

Results And Discussion

Figure 1 shows the FTIR spectra of the unmodified, as well as the phosphated and washed with either NaOH or H₂O avocado seed. It can be seen that the spectrum of the unmodified avocado seed shows the characteristic signals of the cellulose and starch. The signal between 1160 and 1120 cm⁻¹ represents the stretching of the α -(1 $\rightarrow$ 4) and β -(1 $\rightarrow$ 4) glucosidic bonds in the C-O-C groups of starch and cellulose, respectively (Salama et al, 2017, Pratywi et al. 2018). The phosphated avocado seed on the other hand, either washed with NaOH or with H₂O, shows bands close to 860 cm⁻¹, which are attributed to the vibrations of the C-O-P and at 1230 cm⁻¹ which are attributed to the asymmetric stretching of the P = O group (Kaputskii et al. 1988). The sample washed with H₂O (1) shows a signal between 1630 and 1660

cm^{-1} , attributed to the stretching of the N-H and C = O bonds, which is taken as evidence of the presence of urea in the modified avocado sample. The sample washed with NaOH (2), on the other hand, shows no signal between 1630 and 1660 cm^{-1} which is indicative as an absence of urea, which in time indicates that NaOH displaces the urea, replacing it.

Figure 2 (1) shows the proposed chemical structure of the phosphated avocado seed washed with NaOH. The ^{31}P spectrum of the phosphated and washed with NaOH avocado seed in Fig. 2 (2) shows a unique signal at δ 5.22 ppm which corresponds to the phosphorous close to the carbon 6, Fig. 2 (2). In addition, there appears a lack of signals in a negative chemical environment, which discards the formation of pyrophosphates and polyphosphates (Suchyta et al. 2017). Though the structures and discussion presented above correspond to the cellulose in the avocado seed; it is considered that the phosphated starch would present a similar structure, but instead of having β -(1 $\rightarrow$ 4) glucoside bonds of the β -(1 $\rightarrow$ 4) type, these would be of the α -(1 $\rightarrow$ 4) type.

Figure 3 (1) shows the proposed chemical structure of the phosphated avocado seed washed with H_2O . The ^{31}P spectrum of the phosphated and washed with H_2O avocado seed in Fig. 3 (2) shows a unique signal at δ = 0 ppm which corresponds to the phosphorous close to the carbon 6, Fig. 3 (1) "C-O-P" (Hu et al. 2013).

Figure 4 shows the thermograms ran in nitrogen (1) and in air (2), of the unmodified (a) and modified and washed with either NaOH (b) or H_2O (c) avocado seed. Considering the samples ran in nitrogen, it can be observed that the unmodified avocado seed presents a minor weight loss below 100°C, attributed to loss of water, as well as a mayor weight loss between 230 and 340°C, with a maximum rate of weight loss at 290°C. This loss is attributed to the thermal decomposition of the cellulose and starch chains. The phosphated and washed with NaOH avocado seed, also shows the minor weight loss below 100°C attributed to loss of water, as well as a mayor weight loss between 220 and 340°C. However, this mayor weight loss presents two peaks of maximum rate of decomposition. The first, at 240°C is attributed to an acceleration of the polysacharides decomposition, due to the liberation of HPO_3 acid, that acts as the acid soured, promoting the formation of char (Ghanadpour et al. 2015). The second, at 290°C is attributed to the decomposition of the polysacharides that were not dehydrated by the acid source. The phosphated and washed with H_2O avocado seed presents a minor decomposition in two steps. The first, due to loss of water below 100°C and the second, showing two peaks of maximum decomposition rate, at 150 and 200°C. Both correspond to the thermal degradation of urea, which occurs in two steps (Rahmanian et al. 2015). In addition, the second peak also corresponds to the thermal decomposition of some polysacharides. It can also be observed that the residue after heating to 550°C, is 20 wt% for the unmodified avocado seed, whereas it is 42 and 36 wt% for the phosphated and washed with NaOH and H_2O respectively. This is an indication of a higher resistance to thermal decomposition and volatilization due to the phosphating of the avocado seed. Considering the samples ran in oxygen atmosphere, (Fig. 4 (2)), it can be observed that the unmodified avocado seed presents a similar behavior to the one when ran in nitrogen. The one big difference was the almost complete decomposition and vaporization of the

avocado seed, leaving less than 3 wt% residue. However, the phosphated avocado seed, either washed with NaOH or H₂O, left a larger amount of residue, 38 wt% and 28 wt%, respectively, though a little less than the residue in the samples ran in nitrogen. This is an indication of the positive effect of the modification, on retarding the thermal degradation. This is due to the acid source accelerating the dehydration of the polysaccharide chains, promoting the formation of char, which is fire retardant. The difference between the samples washed with NaOH or H₂O is because those washed with NaOH have sodium in its structure, which is fire resistant, whereas those washed with water have urea, which degrades.

Figure 5 presents the SEM image and the elemental analysis of the pulverized and unmodified avocado seed. The pulverized avocado seed shows an ellipsoidal granulated morphology, which is very similar to that of cellulose and starch (Miranda et al. 2017). The EDAX analysis shows that the avocado seed is mainly composed of carbon and oxygen, components of the polysaccharides, plus nitrogen and traces or impurities of magnesium and aluminum.

Figure 6 presents the SEM images and the elemental analysis of the pulverized and phosphated avocado seed, washed with (a) NaOH or (b) H₂O. Samples washed with NaOH show little fibrillar structures, which represent the cellulose or starch, but no agglomerates are observed. Results of the EDAX analysis show the presence of phosphorous from the phosphate modification. Additionally, the absence of nitrogen can be noticed, which indicates that the material is free of urea. A strong peak of sodium is also observed, which indicates the bonding of phosphate and sodium. Samples washed with H₂O show many agglomerates, which occur due to the “packing” between the urea present in the modified avocado seed (Kokol et al. 2015).

The XPS spectra of the unmodified and modified avocado seed is shown in Fig. 7. The unmodified avocado seed shows two peaks at 285.4 and 496.8 eV, which correspond to the elements C 1s and O 1s respectively (Fras et al. 2005). The phosphated and washed with either NaOH or H₂O present the same signals.

The phosphated and washed with H₂O avocado seed presents new signals at 134.4 eV, 191.3 eV and 402 eV, which are attributed to P 2p, P 2s and N 1s respectively (Ding et al. 2016). This confirms the phosphating of the avocado seed and the presence of urea.

The EDAX analysis appear similar to those of samples washed with H₂O; the main difference being the absence of sodium and a greater presence of nitrogen, due to the presence of urea. The samples washed with NaOH, on the other hand, present the peaks that correspond to P 2p and P 2s, but no peak at N 1s is evident. However, new peaks at 48, 63, and 497 eV, which corresponds to Na_{KLL}, Na 2p and Na 2s, respectively. Additionally, there appears a peak at 1072 eV, (which is not shown but it appears when at high resolution (Keshk et al. 2019).

These signals prove the presence of sodium and the substitution of urea, which confirm that the proposed structures are very close approximations of the final structure of the modified avocado seed.

The obtained high resolution XPS spectra were deconvoluted using the Origin Pro 2016 software. The high-resolution O 1 s spectra of unmodified and modified avocado seed (washed with H₂O) are presented in Fig. 8. The spectra of the modified and washed with NaOH is very similar to that washed with H₂O and it is not presented. The unmodified avocado seed (Fig. 8a) shows two peaks, at 532.6 and 531.1 eV which are attributed to COH and COOH respectively. The COH representing 88% of the total peaks whereas the COOH represents 12%. These signals correspond to the polysaccharides that conform the avocado seed (Rouxhet et al. 2008). The modified and washed with H₂O avocado seed (Fig. 8b) also shows two peaks which are also attributed to COH and COOH, with the difference that these peaks overlap the POC and the P = O peaks (Ghanadpour et al. 2015). The COH and POC peak representing 65% whereas the COOH represents 35%. This decrease in the COH peak, compared to the same one in the unmodified sample is because of the reaction between the phosphate and the COH group of the polysaccharides in the avocado seed.

The obtained high resolution XPS spectra were deconvoluted using the Origin Pro 2016 software. The high-resolution C 1 s spectra of unmodified and modified avocado seed (washed with H₂O) are presented in Fig. 9. The spectra of the modified and washed with NaOH is very similar to that washed with H₂O and it is not presented. The unmodified avocado seed (Fig. 9a) shows three peaks, at 284.7, 286.2 and 287.35 eV which are attributed to CC, CO, and OCO respectively, which correspond to the polysaccharides, among which there would be the cellulose and starch of the avocado seed. The amounts corresponding to the CC, CO, and OCO peaks in the unmodified avocado seed are 65, 24, and 11% respectively (Luo et al. 2013).

The modified and washed with H₂O avocado seed (Fig. 9b) also shows three peaks which are also attributed to CC, CO, and OCO, with a difference in the amount that corresponds to each peak.

The high-resolution P 2p spectra of modified avocado seed, washed with NaOH and with H₂O, are presented in Fig. 10. Both present two peaks at ca. 133.7 and 134.5 eV which correspond to P-O-C and PO₃-2 respectively (Zhang et al. 2020). This confirms the phosphating reaction and complements the results obtained in O 1s and C 1s. The amounts corresponding to P-O-C and PO₃-2 would be ca. 65% and 34% respectively, with very slight variations between the one washed with NaOH and the one washed with H₂O. This indicates that the substitution of urea by sodium has no mayor repercussion in the phosphate group of the modified avocado seed. These results also confirm the ³¹P NMR results, since in those spectra with sodium the peak signal shift was in a similar range to that of free phosphoric acid, but these signals prove that there is indeed a chemical bond.

The high-resolution spectra of N 1s of the modified avocado seed washed with H₂O and of Na 1s of the modified avocado seed washed with NaOH, are presented in Fig. 11. The N 1s spectrum of the sample washed with H₂O shows two peaks, at 399.8 and 401.5 eV which correspond to NH₂ and NH₃⁺

respectively (Taylor et al. 2012). These signals are attributed to urea; the NH_2 signal is assigned to the free part of the molecule, whereas the NH_3^+ is assigned to the part bonded to the phosphate. The N 1s spectrum of the sample washed with NaOH, on the other hand, presents a unique signal at 1071.1 eV, attributed to the sodium bonded to the phosphate group (Moulde et al. 2005). This is an indication that the sodium in the sample is ionically bonded and not free sodium, which means that the sodium substitution is possible.

Figure 12 shows the cone calorimeter curves of PE/EVA and three of the studied composites while Table 1 shows the cone calorimeter data obtained from these curves. It can be observed that the pure PE/EVA, burns very rapid and violently. Showing a high heat liberation peak of $1,520 \text{ kW/m}^2$ after 131 s of the test start. The addition of the pulverized unmodified avocado seed also burned very rapid and violently, liberating $1,230 \text{ kW/m}^2$ after 130 s, that is, 81% of the heat liberated by PE/EVA. The addition of pulverized and modified avocado seed produced a marked decrease in the peak of the heat release rate curve, liberating 820 kW/m^2 and 905 kW/m^2 , that is, 54% and 60% of the heat liberated by PE/EVA. The two different values obtained with the modified avocado seed correspond to the modified avocado seed washed with H_2O or with NaOH, respectively. In addition, the combustion is not violent anymore. The time to reach the pHRR decreased, with respect to that of the original sample, to 115 and 105 s, respectively. This is attributed to the liberation of phosphoric acid, which produces a dehydration of the carbon chains and promotes the formation of a protective char layer.

The ttf decreases from 130 to 114 upon addition of unmodified avocado seed, which is simply because the avocado seed is not flame retardant, but on the contrary, being mostly composed of cellulose and starch, they tend to burn and thermally degrade relatively easy. When modified, however, the avocado seed produces an increase in the ttf up to 162 s and 140 s, when using modified avocado seed washed with H_2O or with NaOH, respectively. In addition, the burning process is nonviolent. Finally, the addition of modified avocado seed decreases the THR from 80 to $69 \pm 1 \text{ MW/m}^2$. These results show that the phosphating of the avocado seed produce a marked flame retarding effect.

Photographs showing the char residue of the four different samples examined in the cone calorimeter are presented in Fig. 13. As can be observed, there is no residue of the polymeric sample (a), which burned completely. The polymeric sample with the unmodified avocado seed (b), left a scarce amount of ash, which came from the unmodified avocado seed. The use of modified avocado seed (c) and (d) on the other hand, produced clearly different results. The burning of sample (c) left a significant amount of char that entirely covers the bin. This is due to liberation of phosphoric acid, which promotes the formation of char. The burning of sample (d) also left a significant amount of “clear” char, which comes from the present metallic sodium, which in turn produces the sodium oxides that change the appearance of the char layer (Liu et al. 2020).

Table 1
Cone calorimeter data for PE/EVA blend and Avocado seed biocomposites

Sample	pHRR (kW/m ²)	tpHRR (s)	ti (s)	Te (s)	tff (s)	THR (MW/m ²)
PE/EVA	1520	131	55	185	130	80
PE/EVA AS	1230	130	41	155	114	75
PE/EVA MAS H ₂ O	820	115	38	200	162	70
PE/EVA MAS NaOH	905	105	35	175	140	68

* pHRR = The amount of heat liberated at the peak of the heat release rate curve; tpHRR = The time when the pHRR is reached; ti = the time to ignition, once subjected to a flame; te = The time to extinction of the flame; ttf = Total time of fire (from ignition to extinction) (ttf = te - ti); THR = The total heat released from ignition to extinction of the flame.

Conclusions

The controlled phosphating of the avocado seed, which mostly contains cellulose and starch, was carried out using phosphoric acid in the presence of urea. After phosphating, the hydroxide groups of cellulose and starch are attracted to the phosphate groups of the phosphoric acid. Results showed that the urea chemically bonds to the phosphate groups when the avocado seed is washed with H₂O, but if washed with NaOH, the sodium displaces the urea, producing other final properties. The thermal characteristics of the avocado seed changed markedly after modification with the phosphate groups, this is assumed to be because these groups accelerate the degradation reaction when in the presence of the acid, due to the dehydration of the carbohydrates, which in turn produces the fire resistance char. It was concluded that the presence of sodium increases the amount of residual material, simply because sodium is fireproof, whereas urea degrades even below 250°C. It could be said therefore, that sodium is more effective than urea, because of its thermal stability, but in polymers, the nitrogen could be very useful as a foaming agent for the char intumescent layer, producing thus an intumescent system. The SEM images show that the avocado seed morphology does not change when modified, but urea tends to form agglomerates. The XPS results show that this level of modification may be useful in materials with intrinsically good thermal properties. The cone calorimeter results prove the effectivity and applicability of phosphate avocado seed as bio-filler for polymer systems. A marked reduction in the peak of the heat liberation rate and in the total heat released was observed when tested in PE/EVA composites, as well as a slight increase in the total time of fire. These properties are vital in flame retarding systems. It was also observed that after phosphating, the avocado seed tends to leave a char layer, which combined with intumescent systems may produce highly flame retarding systems. But most important, these results open a door for the use of food waste as specialized vegetable renewable fillers for polymers, without the disadvantage of poor thermal resistance.

Declarations

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Authors' contributions:

J.D Zuluaga-Parra: Experimental work

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J.A. Rodríguez-Gonzalez: Composites preparation and technical support

J.J. Borjas-Ramos: Technical support

S. Vázquez-Rodríguez: XPS analysis

J.A. Uribe-Calderón; Reviewing and writing

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Figures

Figure 1

ATR-FTIR spectra of the pulverized avocado seed; (a) unmodified, (b) phosphated and washed with NaOH, (c) phosphated and washed with H₂O.

Figure 2

Proposed structure of the avocado seed, phosphated and washed with NaOH, (1) ^{31}P NMR spectrum and (2) in D_2O .

Figure 3

Proposed structure of the avocado seed, phosphated and washed with H_2O , (1) ^{31}P NMR spectrum and (2) in D_2O .

Figure 4

TGA-DTG of avocado seed, (a) unmodified, (b) phosphated and washed with NaOH, (c) phosphated and washed with H₂O. In (1) nitrogen and in (2) air.

Figure 5

Morphology and EDAX analysis of pulverized and unmodified avocado seed.

Figure 6

Morphology and EDAX analysis of pulverized avocado seed, phosphated and washed with (a) NaOH or (b) H₂O.

Figure 7

XPS spectra of the pulverized and unmodified, as well as phosphated and washed with either NaOH or H₂O.

Figure 8

High resolution O 1s XPS spectra of the pulverized avocado seed, unmodified as well as phosphated and washed with H₂O.

Figure 9

High resolution C 1s XPS spectra of the pulverized avocado seed, unmodified as well as phosphated and washed with H₂O.

Figure 10

High resolution P 2p XPS spectra of the modified avocado seed, (a) washed with H₂O, as well as Na 1s XPS spectra of the modified avocado seed, (b) washed with NaOH.

Figure 11

High resolution N 1s XPS spectra of the modified avocado seed, (a) washed with H_2O , as well as Na 1s XPS spectra of the modified avocado seed, (b) washed with NaOH.

Figure 12

Cone calorimeter curves showing the variation of the heat release rate (HRR) for the pure PE/EVA, as well as for the polymer with unmodified avocado seed (PE/EVA-AS), and for the polymer with two differently modified avocado seed as filler (PE/EVA-MAS-H₂O and PE/EVA-MAS-NaOH).

Figure 13

Photographs of the char residue after the cone calorimter test, of samples; (a) PE/EVA, (b) PE/EVA-unmodified avocado seed, (c) PE/EVA-modified avocado seed washed with H₂O, and (d) PE/EVA-modified avocado seed washed with NaOH.