SM/EB -16

Changes in Physicochemical, Morphological and Thermal Properties of Electron-beam Irradiated Ethylene–Vinyl Alcohol Copolymer (EVOH) as a Function of Radiation Dose

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We Studied the Changes in these properties

of EVOH resin and EVOH resin reinforced with piassava fiber after electron-beam irradiation

… and the correlation between properties of the EVOH resin and EVOH with piassava fiber

EVOH Copolymers

a family of resins with ➔

- superior gas barrier properties compared to most of the polymeric materials;
- excellent gas barrier properties to oxygen and organic compounds;
- low absorption rate of odor and flavor;
- excellent resistance to oils and organic solvents;
- good chemical resistance;
- high transparency and
- easy processability.

EVOH Copolymers - Application

are widely used in various fields such as:

- food packaging;

- organic solvents packaging;

EVOH Copolymers - Application

- agricultural chemicals packaging;

- gasoline tanks

- and others.

EVOH Copolymers - Challenges

they are very sensitive to moisture,

high relative humidity conditions
due to water absorption

- their gas barrier ability deteriorates and
- their thermal and mechanical properties are affected too.

Piassava

(Attalea Funifera Mart.)

lignocellulosic fiber extracted from the leaves of a palm tree native to the Brazilian Atlantic rainforest

Piassava

(Attalea Funifera Mart.)

- it has higher lignin content than any other lignocellulosic fiber
- lignin could be responsible for its inherent flexural rigidity and water proof resistance

Piassava

(Attalea Funifera Mart.)

50% of the fiber is disposed as residue by the transformation industry

- 30% is discarded during the cut, cleaning and baling
- 20% is discarded by brooms, brushes, ropes, baskets, and carpets manufacturers, before production.

Experimental

Material:

- EVOH resin containing 68 mol% ethylene and
- Piassava (Attalea funifera Mart) fiber residues (disposed by some brooms and brushes manufacturers)

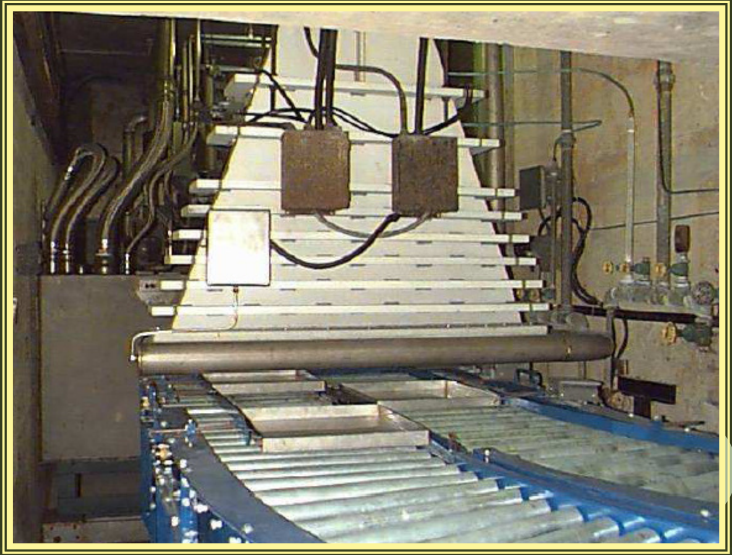
Piassava fiber ➔ after washed and dried ➔ reduced to fine powder using ball mills ➔ particle sizes ≤ 200 µm.

EVOH resin reinforced with 10% of piassava fiber (in weight) ⬇ by using a double screw extruder machine.

EVOH and EVOH-piassava

↕ e-beam Irradiation

- electrostatic accelerator (1.5MeV)
- radiation dose up to 90 kGy
- dose rate 11.22 kGy/s
- room temperature
- in air



Analyses

- differential scanning calorimetry (DSC)
- thermogravimetric analysis (TGA),
- scanning electron microscopy (SEM) and
- sol-gel analysis.

Results and Discussion

Differential scanning calorimetry (DSC)

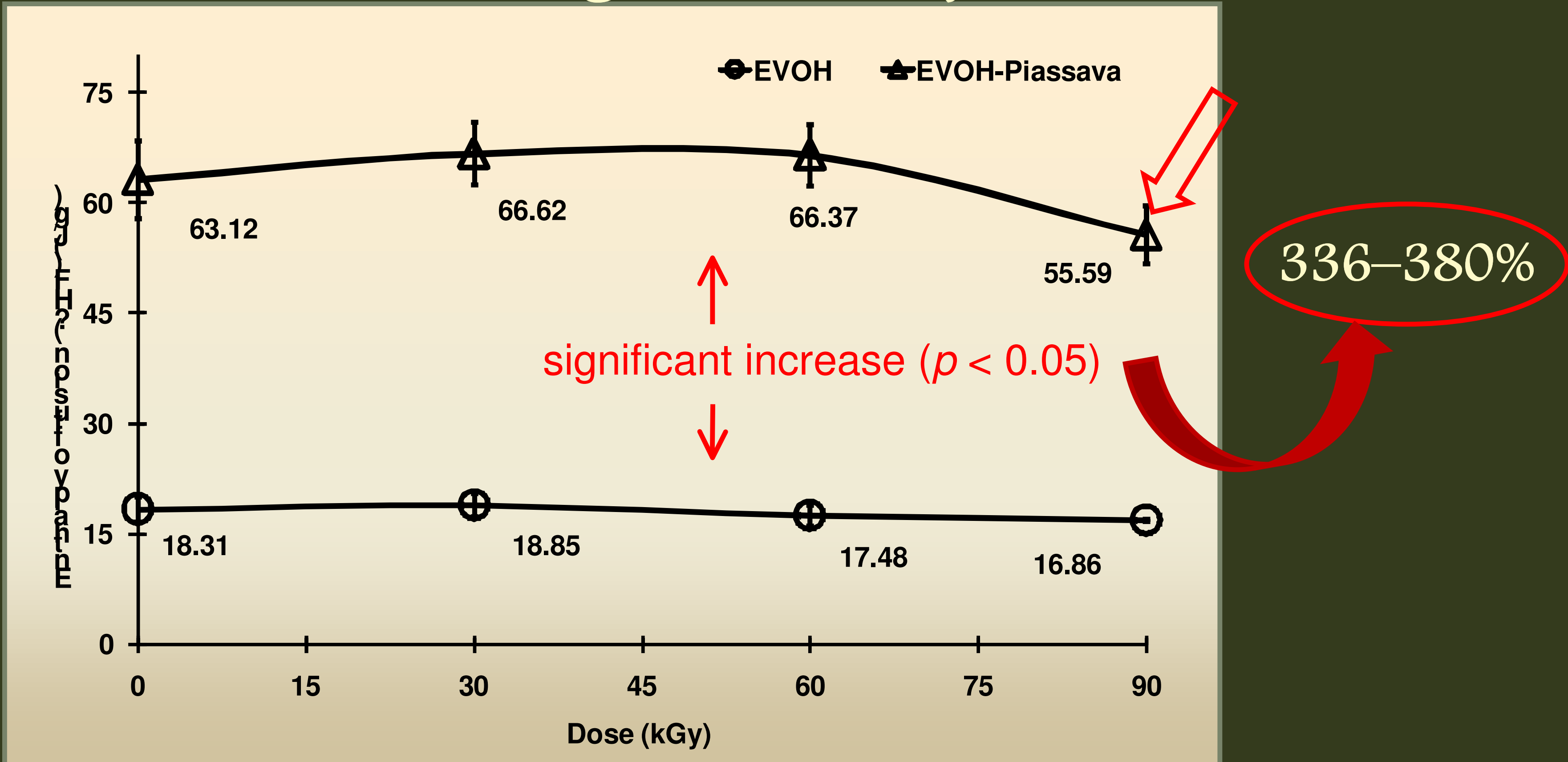


Figure 1. Effects of irradiation on melting enthalpy (ΔH_m) as a function of electron-beam radiation dose for the EVOH and EVOH-piassava.

Table I. Crystallinity percentage variation as a function of electron-beam radiation dose

DOSE (kGy)	EVOH[a]			EVOH-piassava[b]			EVOH-piassava/EVOH[c]
	ΔH_m (J/g)	$\Delta\tau$ statistic differences ($p < 0.05$)	$\Delta\tau$ (%)	ΔH_m (J/g)	$\Delta\tau$ statistic differences ($p < 0.05$)	$\Delta\tau_p$ (%)	$\Delta\tau_p$ (%)
0	18.31		-	63.12		-	71.00
30	18.85	ns[d]	-	66.62	ns[d]	-	69.72
60	17.48	ns[d]	-	66.37	ns[d]	-	73.66
90	16.86	ns[d]	-	55.59	s[e]	11.93	70.18

[a] crystallinity percentage variation EVOH as a function of electron-beam radiation dose; [b] crystallinity percentage variation EVOH-piassava as a function of electron-beam radiation dose; [c] crystallinity percentage variation between EVOH-piassava and EVOH; [d] non-significant; [e] significant.

↑ EVOH –piassava crystallinity ↑ barrier ↓ Water absorption rate

Thermogravimetric Analysis (TGA)

Table II. Initial degradation temperature and weight loss of the EVOH and EVOH-piassava as a function of electron-beam radiation dose

Dose (kGy)	EVOH			EVOH-piassava			EVOH-piassava/ EVOH
	T_i [a] (°C)	T_f [b] (°C)	Weight Loss (%)	T_i [a] (°C)	T_f [b] (°C)	Weight Loss (%)	T_i Difference[c] (%)
0	370.60	412.13	76.79	378.67	430.75	85.91	2.18 s[e]
30	367.29 ns[d]	416.08 ns[d]	75.52 ns[d]	379.41 ns[d]	429.16 ns[d]	85.07 ns[d]	3.30 s[e]
60	368.40 ns[d]	425.71 ns[d]	77.87 ns[d]	379.70 ns[d]	438.03 s[e]	84.62 ns[d]	3.07 s[e]
90	368.23 ns[d]	425.58 ns[d]	77.65 ns[d]	378.61 ns[d]	434.26 s[e]	83.08 ns[d]	2.82 s[e]

[a] initial degradation temperature; [b] final degradation temperature; [c] difference of initial degradation temperature between EVOH and EVOH-piassava; [d] statistically non-significant ($p < 0.05$); [e] statistically significant ($p < 0.05$).

Scanning Electron Microscopy (SEM)

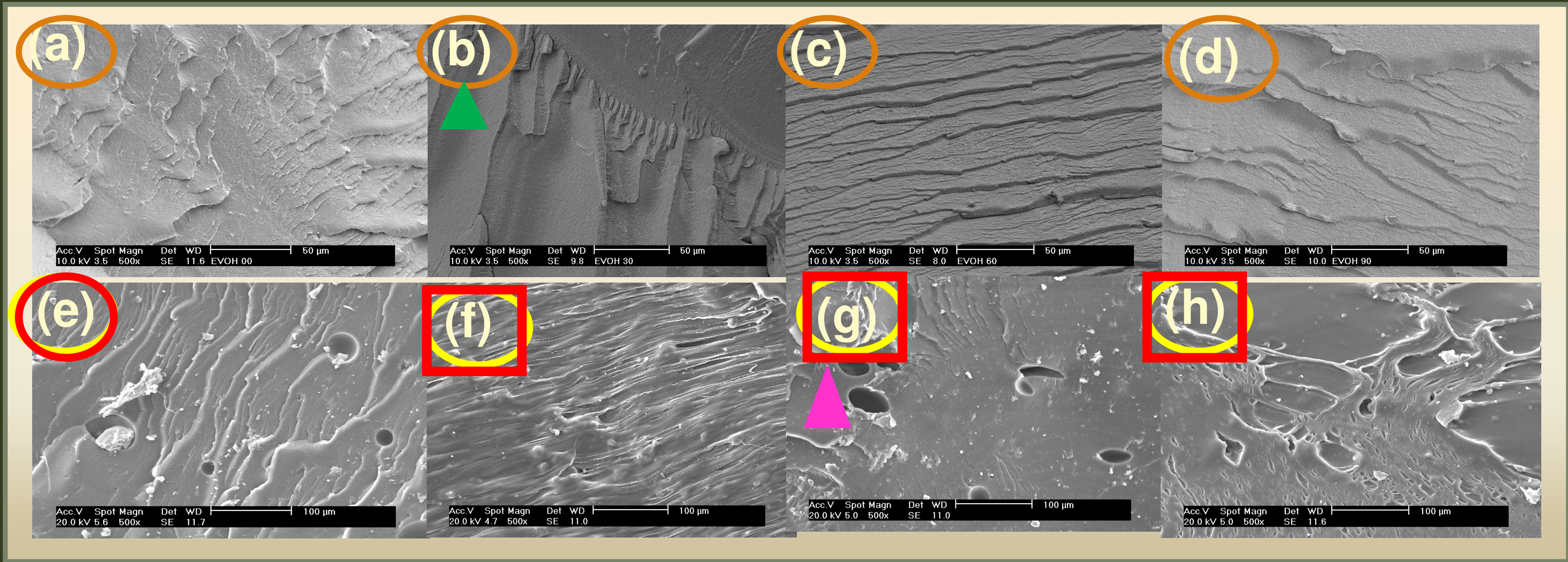


Figure 2. Scanning Electron Microscopy (SEM) micrographs for EVOH and EVOH-piassava at ranges electron-beam radiation dose studied. Fig. 2(a) non-irradiated EVOH; Fig. 2(b) EVOH at 30 kGy; Fig. 2(c) EVOH at 60 kGy; Fig. 2(d) EVOH at 90 kGy; Fig. 2(e) non-irradiated EVOH-piassava; Fig. 2(f) EVOH-piassava at 30 kGy; Fig. 2(g) EVOH-piassava at 60 kGy; Fig. 2(h) EVOH-piassava at 90 kGy.

EVOH ➔ rough, dense and compact, 30 kGy ➔ slightly smoother, ↑ brittle

EVOH-piassava ➔ 2 phases ➔ dispersed phase (piassava particles); EVOH continuous phase

Sol-Gel Analysis

The results did not show gel content in samples after extraction with solvent.

electron-beam radiation at radiation dose applied in this work was not enough for cross-linking the EVOH and EVOH-piassava materials.

Conclusions

The ΔH_m of EVOH and the ΔH_m of EVOH-piassava basically was not affected by irradiation, except for EVOH-piassava at 90 kGy

Electron-beam radiation doses applied promoted better interfacial adhesion between piassava fiber and EVOH resin

Conclusions

Compared to original EVOH,

EVOH-piassava presented:

- a large difference in crystalline form

  ↳ order of macromolecules arrangement of EVOH was changed by piassava incorporation

- higher crystallinity > 70-74 % ⇨EVOH resin
- higher initial degradation temperature > 3%

Conclusions

These results are very important because:

They could lead to the obtaining of materials with...

- better barrier properties
- lower water absorption rate
- more stable thermal and mechanical properties

… in high relative humidity conditions than original EVOH resin

THANK YOU FOR YOUR ATTENTION

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