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Poly lactide films produced with bixin and ATBC: effect of thermal treatment on bixin stability and migration kinetic

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1. Introduction

The incorporation of antioxidants into a polymeric structure to produce antioxidant active materials is an alternative way to extend the shelf-life of fatty food products. Even though polylactide (PLA) present satisfactory mechanical properties, it requires improvements to be used as an antioxidant material for food packaging.

Bixin, a carotenoid with hydrophobic character, presents coloring and antioxidant properties and hence is a potential antioxidant additive for PLA. Considering bixin thermal low stability, films produced with bixin and the plasticizer acetyl tributyl citrate (ATBC) were produced by different thermal processes. The effect of thermal treatments over films color changes and bixin stability was evaluated, also as its effect on bixin's kinetic of migration.

2. Materials and Methods

Bixin was extracted from annatto seeds (*Bixa orellana*) with dichloromethane. Polylactide (NaturePlast PLE 001) was used to develop the active films, where two formulations (F-without plasticizer; A-with plasticizer) were produced by different methods. Formulations and methods are shown below.

Table 1. PLA films formulations and method of obtention

	Bixin (%)	ATBC (%)	Film Processing Method
F_cast	0.1	0	casting
A_cast	0.1	13	casting
F_TP	0.1	0	casting + TP (6 min)
A_TP	0.1	13	casting + TP (6 min)
F_IMTP	0.1	0	IM (2 min) + TP (4 min)
A_IMTP	0.1	13	IM (2 min) + TP (4 min)

TP: thermo-press (160°C/5 tons); IM: internal mixer (160 °C, 50 rpm)

The **bixin loss** due to thermal processing of films was evaluated by films dissolution in dichloromethane, followed by bixin quantification by spectroscopy ($\lambda=467$ nm; $A_{1\%}=2826$).

The effect of the thermal treatments over films **color stability** was evaluated using a Konica Minolta colorimeter through L*(luminosity), a* (red-green), and b* (yellow-blue) parameters. The total color differences (ΔE) caused by thermal processes when compared to casting films was calculated using the following equation

$$\Delta E = \sqrt{(L^* - L^*_{cast})^2 + (a^* - a^*_{cast})^2 + (b^* - b^*_{cast})^2} \quad (1)$$

where L*, a* and b* were measured in TP and IMTP films.

Bixin **migration kinetic** to ethanol 95% v/v (used as a fat food simulant) was evaluated over 65 days at 40 °C. Quantification was made by spectroscopy and the **overall mass transfer coefficient (K)** of bixin was obtained using the following equation:

$$\frac{dC}{dt} = K \frac{A}{V} (C^* - C) - k_d C \quad (2)$$

C: bixin concentration in the food simulant ($\mu\text{g mL}^{-1}$);
 C*: bixin concentration ($\mu\text{g mL}^{-1}$) in the inner part of the film;
 K: overall mass transfer coefficient (m s^{-1});
 A: surface area of films;
 V: volume of food simulant;
 k_d : first-order kinetic degradation coefficient of bixin in ethanol 95% at 40 °C ($2.155 \text{ s}^{-1} \times 10^{-7}$)
 (Stoll et al., 2019)

3. Results and Discussion

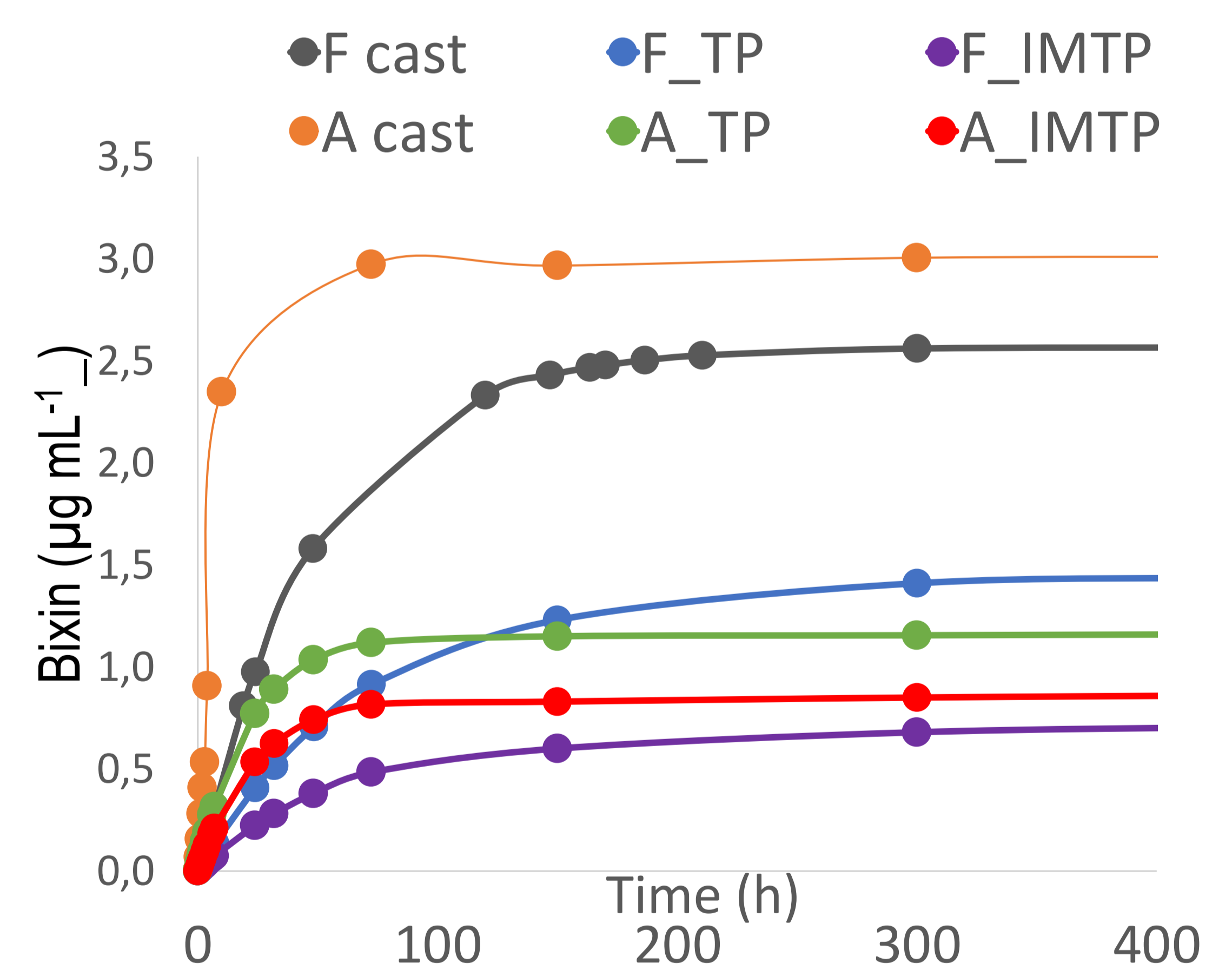
Table 2. Color parameters, bixin loss and bixin overall migration rate (K) of PLA films

	L*	a*	b*	ΔE	Bixin loss (%)	K (m s^{-1})
F_cast	79.17	13.14	87.10	-	-	1.6 c
A_cast	80.47	9.51	89.36	-	-	5.8 ab
F_TP	83.67	2.88	105.50	21.5 b	74.5 b	2.1 c
A_TP	84.88	-1.54	103.01	18.2 b	85.3 a	4.4 b
F_IMTP	80.62	6.00	99.86	39.9 a	79.9 a	7.1 a
A_IMTP	81.94	5.75	103.83	39.4 a	79.2 a	6.9 a

Different letters in the same column indicate significant differences ($p < 0.05$) among the treatments

- ✓ A decrease in films redness (reduction in a*) and an increase in yellowness (increase in b*) occurred due to bixin thermal degradation, where films produced by **IMTP presented the highest change in color** ($\Delta E \approx 40$). Despite the color changes, films presented a strong and vibrant orange-yellow color after the thermal treatments.
- ✓ The presence of **ATBC increased in 10% the bixin loss** in TP films. Considering PLA is frequently used on the encapsulation of diversal bioactive compounds – presenting a protective effect - its replacement by ATBC might have reduced bixin stability in films matrix.
- ✓ The presence of **ATBC greatly increased bixin migration rate** from casting and TP films, which has been ascribed to the increased polymer chain mobility caused by the plasticizer, favoring the active compound release.

Figure 1. Bixin migration from films to ethanol 95% (v/v)



- ✓ **IMTP films** presented the **highest overall mass transfer coefficient (K)** of bixin to the food simulant. The cleavage of PLA long chains caused by internal mixer shear forces might facilitate bixin diffusion through the polymeric matrix, increasing its release rate to the food simulant.

4. Conclusion

The presence of ATBC and mixing shear forces affected bixin stability and migration rate. Since antioxidants' concentration and release rate greatly affect the performance of antioxidant active packaging, these findings are of great importance and are currently being explored on the extension of fatty food products shelf-life.

5. References

Stoll et al. Poly(acid lactic) films with carotenoids extracts: Release study and effect on sunflower oil preservation. Food Chem 281 (2019) 213–221