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Antioxidant compounds from blackberry *(Rubus fruticosus)* pomace: Microencapsulation by spray-dryer and pH stability evaluation☆

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ABSTRACT

This study aimed to extract antioxidant compounds from blackberry pomace using two solvents, microencapsulate them by spray dryer and evaluate their stability at different pHs. Two pure extracts (aqueous and hydroalcoholic) and two microencapsulated with maltodextrin were dried in spray dryer and analyzed for stability under different pHs with respect to color variation (ΔE), total phenolic compounds, anthocyanins, antioxidant activity, and kinetic degradation of anthocyanins in 7 days. Regarding the color, the microcapsules protected the samples. Microencapsulation with maltodextrin was efficient in reducing the degradation of anthocyanins against increaesed pH, greater stability of the encapsulated samples was observed in lower pHs, as well as longer half-lives of anthocyanins, values from 2 to 7 times greater when compared to extracts. The use of water in the extraction proce'ss resulted in lower percentage loss in 7 days, which was advantageous in relation to other types of extractions using organic solvents.

1. Introduction

Wild fruits, such as the blackberry (*Rubus fruticosus*), are popular for the combination of their pleasant color and taste, as well as for the reported health benefits for humans (D'Agostino et al., 2015). Some studies report a relation between antioxidant activity and the presence of phenolic compounds and anthocyanins in blackberries (Rosa et al., 2014). Residues originated by the processing industry of blackberries generate around 20% of peel and seeds (Ignat, Volf, & Popa, 2011).

Phenolic compounds contribute in reduction of risks for degenerative diseases, and their effects on human health have been mainly attributed to their antioxidant activity (Machado, Pasquel-Reátegui, Barbero, & Martínez, 2015; Sariburun, Şahin, Demir, Türkben, & Uylaşer, 2010). In addition, anthocyanins are natural water-soluble dyes, responsible for the typical color of blackberries, and have been considered as potential substitutes for synthetic dyes in the food industry (Haminiuk, Maciel, Plata-Oviedo, & Peralta, 2012; Li et al., 2012).

Regarding stability, the main disadvantage of anthocyanins against synthetic dyes is the change in color due to chemical reactions with food, since this pigment has chromophoric groups that are very sensitive to changes in pH (Lopes, Xavier, Quadri, & Quadri, 2007).

In acid pH anthocyanins are in the form of flavilium cations of red coloration, with an increase in pH a deprotonation occurs forming a blue quinoidal base. Anthocyanins may also be present in the pseudo-base carbitol forms which are colorless, and mildly yellowish chalcone (Heredia, Francia-Aricha, Rivas-Gonzalo, Vicario, & Santos-Buelga, 1998).

Extraction of antioxidant compounds such as phenolics and anthocyanins is usually carried out with the aid of organic solvents, with stirring or heating, however it can not be considered a clean technology. Extraction with water is a viable alternative to be used in the food industry, being more economic and environmentaly safe (Ivanovic et al., 2014; Reátegui, Machado, Barbero, Rezende, & Martínez, 2014).

Some techniques may improve stability of extracted compounds during storage, such as microencapsulation, defined as a process in which small particles or droplets are surrounded by a coating, or incorporated into a homogeneous or heterogeneous matrix, resulting in small capsules with many useful properties (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007). Several types of coatings can be used in this process, and carbohydrates, such as maltodextrins, have been widely used as encapsulating agents (Cano-Higuita et al., 2015; Goula & Adamopoulos, 2012).

Among the main desirable characteristics to microencapsulating agents are low hygroscopicity, low viscosity at high solids concentration, ability to emulsify and stabilize the core material, non-reactivity,

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good film formation, maximum protection against light, pH and oxygen, absence of unpleasant taste and odor, and low cost (Cano-Higuita et al., 2015; Shahidi & Han, 1993).

It is worth noting that future tests may be interesting to indicate the application of the dye, and the sensorial tests considering a descriptive and hedonic approach, are interesting to replace industrial dyes by natural dyes in food products (Oliveira et al., 2017; Torres et al., 2017).

Some studies evaluated the action of jabuticaba skin extract in lactic products, as a probiotic petit suisse cheese, compared with another antioxidants. The addition of the jabuticaba extract was able to mantain probiotic culture counts and high antioxidant activity (Pereira, Cavalcanti et al., 2016; Pereira, Faria et al., 2016).

The objective of this study was to produce microcapsules from blackberry pomace using hydroalcoholic and aqueous extraction and evaluating the stability of the samples at different pHs.

2. Material and methods

2.1. Materials

Blackberry (*Rubus fruticosus*) pomace used in this research was purchased from one batch from a producer of Paraibuna city in the state of São Paulo. Maltodextrin (DE10) was supplied by Cargil® (Campinas-SP). The other reagents used were of analytical grade.

2.2. Extraction and microencapsulation of bioactive compounds

Capsules and extracts were dried under the same conditions. Dry aqueous extract by spray dryer (EA) is the blackberry pomace diluted in water at a concentration of 500 mg/mL and mechanically stirred at 60 °C for 45 min, according to an experimental design conducted to evaluate the influence of temperature and time on anthocyanin extraction. Dried hydroalcoholic extract in spray dryer (EE) is the blackberry pomace diluted in the same ratio as above using 80% (v/v) ethyl alcohol under mechanical stirring for 48 h, filtered and rotated at 65 °C until total evaporation of the solvent (Shirahigue et al., 2011), afterwards the samples were filtered and dried in a spray dryer.

Two microcapsules were made, one from the aqueous extract, encapsulated with maltodextrin (CA), where the blackberry residue was diluted in water at 500 mg/mL, shaken for 45 min at 60 °C and filtered, the carrier agent Maltodextrin DE 10 was added directly to the filtrate by mechanical stirring (1: 1 w/w) (Ferrari, Germer, Alvim, Vissotto, & de Aguirre, 2012), and the other from the hydroalcoholic extract encapsulated with maltodextrin (CE), where the residue was diluted in ethyl alcohol 80% (v/v) under mechanical stirring for 48 h, filtered and rotated at 65 °C until total solvent evaporation (Shirahigue et al., 2011), subsequently the carrier agent maltodextrin DE 10 was added (1: 1 w/w) (Ferrari et al., 2012).

The samples EA, EE, CA and CE were submitted to spray dryer (Valduga, Lima, Do Prado, Ferreira Padilha, & Treichel, 2008), under the conditions: inlet drying air temperature 170 °C and outlet 105 °C; Atomization pressure: 4 bar; Average drying air flow: 3.5 m3/h; Average feed rate: 0.5 L/h in Buchi B-191 Mini Spray-dryer equipment. The dried products were placed in plastic bags and stored in a freezer.

2.3. pH estability

Solutions containing the samples (0.625 mg/mL) were prepared using buffers at pHs 2, 3.5, 5 and 6.5. Citric acid and sodium phosphate were used in the preparation of *Mcllvaine* buffer solutions (Morita & Assumpção, 1995), to those 5% of potassium sorbate was added in order to avoid contamination by microorganisms.

Sample stability in the solutions was evaluated for anthocyanins, phenolic compounds, antioxidant activity and color, at different pHs, at the initial time (t0) and after seven days (t7) at constant temperature of 25 °C.

2.4. Total phenolic compounds determination

The determination of total phenolic compounds (TPC) was performed using Folin-Ciocalteu reagents (50%) and sodium carbonate 3.79 M (Na_2CO_3) (Pierpoint, 1996; Singleton & Rossi, 1965). The absorbance was verified in an spectrophotometer at 725 nm after 30 min of incubation at 25 °C. Gallic acid was used as the standard for calibration curve. The results were expressed in μg of gallic acid equivalent (GAE) mg^{-1} of product.

2.5. Determination of the total monomeric anthocyanins content

The differential pH method (Lee, Durst, & Wrolstad, 2005) was used to determine total monomeric anthocyanins. The results were expressed in μg cyanidin-3-glucoside.mg^{-}1, according to Eqs. (1) and (2).

$$AT = (ABS520nm - ABS700nm)pH1.0 - (ABS520nm - ABS700nm)pH4.5 \quad (1)$$

$$Anthocyanins(ACN) = \left[\frac{(AT \times PM \times 10^3)}{\varepsilon \times \lambda}\right] \div C \quad (2)$$

Where: PM = 449.2 g/ mol (molar mass of cyanidin-3-glucoside); 10^3 = conversion factor from g to mg; Ɛ = 26900L/mol (molar absorptivity of cyanidin-3-glucoside); ƛ = 1 cm (optical length of the cuvette). C = sample concentration

2.6. Antioxidant activity by the radical sequestration method DPPH˙ (2,2-diphenyl-1-picrylhydrazine)

The reduction of the stable radical DPPH (2,2-diphenyl-1-picrylhydrazyl) was determined by spectrophotometer colorimetry (Thaipong, Boonprakob, Crosby, Cisneros-Zevallos, & Hawkins Byrne, 2006). The absorbance was verified in an spectrophotometer at 515 nm after 1 h of incubation at 25 °C, the efficiency of the sequestering activity was calculated according to Eq. (3).

$$Efficience\ of\ free\ radical\ sequestration\,(\%) = \frac{(A_{control} - A_{sample})}{A_{control}} \times 100 \quad (3)$$

Where: A Control: Absorbance of negative control; A Sample: sample absorbance average.

The results were expressed as equivalent antioxidant capacity (EC50) μg.mL product^{-1} calculated by the Graphpad Prism 5 software.

2.7. Color analysis

The color was evaluated by a Minolta® CR400 portable colorimeter, with an integration sphere and view angle of 3°, that is, illumination d/3 and illuminant D65. The system used was CIEL *a *b *.

Color difference values (ΔE) were calculated according to Eq. (4) (Obón, Castellar, Alacid, & Fernández-López, 2009).

$$\Delta E = [(L_7^* - L_0^*)^2 + (a_7^* - a_0^*)^2 + (b_7^* - b_0^*)^2]^{0,5} \quad (4)$$

Where: L^*_0, a^*_0 e b^*_0 are the values of the samples at time zero and L^*_7, a^*_7 e b^*_7 the values after 7 days of storage.

2.8. Loss percentage

The percentage (%) loss of anthocyanins and phenolic compounds in the storage period was calculated by the relation between the quantity at the last storage day (t7), by the initial quantity (t0) according to Eq. (5) (de Souza et al., 2014).

$$Totalloss\,(\%) = \left(1 - \frac{t7}{t0}\right) \times 100 \quad (5)$$

2.9. Anthocyanin degradation

Anthocyanin degradation was calculated by Eq. (6), where C is the remaining concentration; C_0 is the initial concentration; T is the time interval between C_0 and C; K is the first-order transformation constant (1/time).

$$\frac{dC}{dt} = -k.\,C \quad (6)$$

Where: C is the remaining concentration; C_0 is the initial concentration; T is the time interval between C_0 and C; K is the first-order transformation constant (1/time).

In a plot of concentration ($-\ln C$) versus time, the transformation constant (k) is the slope of the line.

The half-life (t1/2) for the reaction is the time required for the amount of anthocyanins to fall by half of their initial value. It is directly related to the velocity constant for a first order reaction described by Eq. (7) (Kırca & Cemeroğlu, 2003).

$$t_{1/2} = \frac{\ln(2)}{k} \quad (7)$$

2.10. Statistical analysis

The results were submitted to variance analysis and Tukey's test for the minimum significant difference ($p < 0,05$) between averages using the statistical program Statistica version 7.0.

3. Results and discussion

Fig. 1 shows color variation (ΔE) of the samples in different pHs. EA and EE extracts presented higher color variations at pH 2.0, 3.5 and 5.0 when compared to CA and CE (Fig. 1), indicating that microencapsulation resulted in greater color stability (L*, a* and b*).

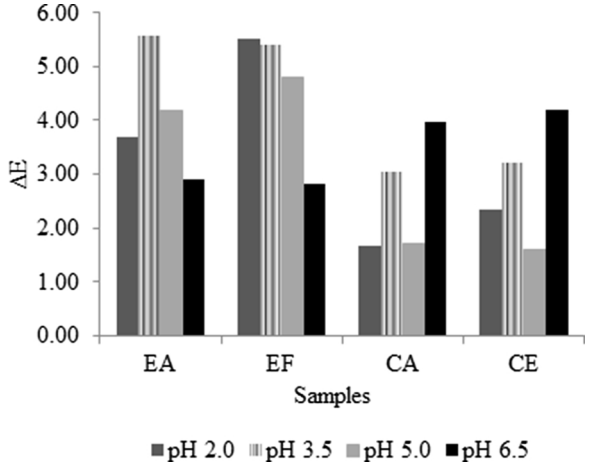

Fig. 1. Solution color variation (ΔE) with samples at the different pHs (2.0, 3.5, 5.0 and 6.5).

Evaluating the capsules in relation to pH, for CA and CE lower color variations were observed at pHs 2.0 and 5.0, indicating higher anthocyanin stability at lower pHs (Türker & Erdoğdu, 2006). The lower color variation at pH 5.0 is important for food application since there is a great range of foods at this pH.

Considering the time of storage evaluated, significant variations are found in values of ΔE greater than 5.0, thus it was observed that the microcapsules maintained color better during the storage time when compared to the extracts (Obón et al., 2009).

Table 1 presents the results of total phenolic compounds, anthocyanins and antioxidant activity of samples in different pHs. At pH 2.0 total phenolic compounds increased in all samples during 7 days of storage, the same relation was observed in the other pHs for EA, CA and CE samples. However, only EE sample was degraded at pH 3.5, 5.0 and 6.5. The increase in TPC may be related to the degradation of anthocyanins in other phenolic compounds, such as coumarin 3,5-diglycoside (Damodaran, Parkin, & Fennema, 2010).

Total monomeric anthocyanins were maintained in the samples EA, EE, CA, CE at the lowest pHs (2.0 and 3.5), and showed more stability than in the other pHs studied.

Degradation of 77.69% of anthocyanins in EA, t7 was observed at pH 2.0, and 78.54% in EE. Degradation was much lower in the microcapsules, 27.72% in CA and 36.36% in EC. Degradation was gradually higher as pH increased, at pH 6.5 the degradation was 100% in all samples.

pH influenced the degradation of anthocyanins, increasing pH may result in reduced pigment stability (Türker & Erdoğdu, 2006). These can undergo reactions that alter their structures due to the deficiency of their flavilium nuclei. The stability of anthocyanins increases with the number of methoxyls and decreases as hydroxyls increase, thus anthocyanins are more stable at acid pH (Hou, Qin, Zhang, Cui, & Ren, 2013).

The antioxidant activity was influenced by pH, no significant difference was observed in pHs 2.0 and 3.5 during 7 days of storage. With increasing pH (5.0 and 6.5), there was reduction of anthocyanins present in the samples, an overall reduction of antioxidant activity was observed. This fact can be explained by the degradation of anthocyanins that generates phenolic compounds with lower antioxidant activity, thus justifying the increase of TPC and the reduction in antioxidant activity with increasing pH. As reported in literature, the antioxidant activity of berries is strongly related to phenolic compounds, as found in other studies (Garcia-Mendoza et al., 2017; Machado et al., 2015).

In relation to the type of extraction used, CA had the lowest loss percentages in 7 days at pHs 2,0, 3,5 and 5,0, proving the advantage of water as a solvent for extraction for its low cost compared to EC using organic solvent.

Fig. 2 shows the values of half-life and the degradation constant of the samples. It was observed that the anthocyanin degradation obeyed a first order kinetics, as reported in literature (Kırca & Cemeroğlu, 2003).

At pHs 2.0, 3.5 and 5.0 the microcapsules $t_{1/2}$ for CA and CE were at least 2 to 7 times higher when compared to extracts EA and EE, the highest $t_{1/2}$ was found at the lowest pH for the microcapsules, indicating the protection of anthocyanins by encapsulation at these pHs.

The half-life was gradually reduced in relation to increase in pH, varing from 14 days at pH 2.0 to 0.8 days at pH 6.5, as the degradation constants are inversely proportional to the half-life, the lowest constants were found at the lowest pHs. Research that evaluates the microencapsulation of blackberry pomace is scarce, so the stability, as well as the degradation parameters in relation to pH have not yet been reported.

Table 1
Total phenolic compounds, total monomeric anthocyanins and antioxidant activity (EC50) of solutions at initial (t0) and final (t7) time.

pH 2.0

	CFT (μg GAE mg produto)		ACN (μg cianidin-3-glucoside mg^{-1})		EC_{50} (μg mL $produto^{-1}$)	
	t_0	t_7	t_0	t_7	t_0	t_7
EA	$52.53^{bB} \pm 0.00$	$66.62^{bA} \pm 2.53$	$1.21^{cA} \pm 0.00$	$0.27^{dB} \pm 0.05$	$44.33^{cA} \pm 0.58$	$43.67^{cA} \pm 1.15$
EE	$91.80^{aB} \pm 0.85$	$103.20^{aA} \pm 0.48$	$2.47^{aA} \pm 0.01$	$0.53^{cB} \pm 0.05$	$23.00^{dA} \pm 0.00$	$21.50^{dA} \pm 0.58$
CA	$23.61^{dB} \pm 0.18$	$29.47^{dA} \pm 0.81$	$1.01^{dA} \pm 0.01$	$0.73^{bB} \pm 0.04$	$91.67^{aA} \pm 1.53$	$94.33^{aA} \pm 2.52$
CE	$26.34^{cB} \pm 0.08$	$37.07^{cA} \pm 0.53$	$1.98^{bA} \pm 0.04$	$1.26^{aB} \pm 0.01$	$65.67^{bA} \pm 0.58$	$66.00^{bA} \pm 5.20$

pH 3.5

	CFT (μg EAG mg produto)		ACN (μg cianidina-3-glucosídeo mg^{-1})		EC_{50} (μg mL $produto^{-1}$)	
	t_0	t_7	t_0	t_7	t_0	t_7
EA	$48.62^{bB} \pm 1.59$	$61.29^{bA} \pm 3.25$	$0.89^{bA} \pm 0,00$	nd	$44.00^{cA} \pm 0.00$	$46.67^{cA} \pm 2.52$
EE	$87.91^{aA} \pm 2.40$	$79.16^{aB} \pm 2.82$	$2.07^{aA} \pm 0.00$	$0.21^{cB} \pm 0.01$	$28.50^{dA} \pm 1.53$	$31.67^{dA} \pm 2.08$
CA	$22.24^{cB} \pm 0.43$	$30.51^{dA} \pm 1.00$	$0.88^{bA} \pm 0.04$	$0.31^{bB} \pm 0.00$	$95.50^{aA} \pm 9.07$	$98.67^{aA} \pm 0.58$
CE	$24.96^{cB} \pm 0.92$	$37.56^{cA} \pm 2.34$	$1.97^{aA} \pm 0.06$	$0.67^{aB} \pm 0.01$	$71.50^{bA} \pm 3.21$	$78.00^{bA} \pm 2.52$

pH 5.0

	CFT (μg EAG mg produto)		ACN (μg cianidina-3-glucosídeo mg^{-1})		EC_{50} (μg mL $produto^{-1}$)	
	t_0	t_7	t_0	t_7	t_0	t_7
EA	$52.27^{bA} \pm 1.35$	$53.64^{bA} \pm 1.24$	$0.42^{cA} \pm 0.01$	nd	$46.50^{cA} \pm 0.58$	$50.00^{cA} \pm 3.61$
EE	$84.18^{aA} \pm 0.78$	$75.73^{aB} \pm 0.81$	$1.61^{aA} \pm 0.05$	nd	$28.00^{dB} \pm 0.58$	$34.67^{dA} \pm 0.58$
CA	$21.29^{dB} \pm 0.70$	$25.62^{dA} \pm 0.38$	$0.65^{bA} \pm 0.04$	$0.17^{bB} \pm 0.01$	$174.00^{aA} \pm 0.58$	$158.50^{aB} \pm 6.36$
CE	$24.21^{cB} \pm 0.58$	$37.80^{cA} \pm 3.24$	$1.63^{aA} \pm 0.01$	$0.33^{aB} \pm 0.01$	$72.33^{bB} \pm 2.31$	$100.50^{bA} \pm 7.78$

pH 6.5

	CFT (μg EAG mg produto)		ACN (μg cianidina-3-glucosídeo mg^{-1})		EC_{50} (μg mL $produto^{-1}$)	
	t_0	t_7	t_0	t_7	t_0	t_7
EA	$50.40^{bB} \pm 0.53$	$53.16^{bA} \pm 1.37$	$0.1^{dA} \pm 0.01$	nd	$52.00^{cB} \pm 1.41$	$93.00^{abA} \pm 4.24$
EE	$82.76^{aA} \pm 2.01$	$75.16^{aB} \pm 1.69$	$1.26^{bA} \pm 0.01$	nd	$52.00^{cA} \pm 1.41$	$65.00^{bA} \pm 19.80$
CA	$24.03^{cA} \pm 3.20$	$23.22^{dA} \pm 0.67$	$0.55^{cA} \pm 0.01$	nd	$97.00^{aA} \pm 7.51$	$105.67^{aA} \pm 6.66$
CE	$25.94^{cB} \pm 0.76$	$30.87^{cA} \pm 1.40$	$1.37^{aA} \pm 0.02$	nd	$72.50^{bB} \pm 3.79$	$103.00^{abA} \pm 2.83$

Averages followed by the same lowercase letter in the column and upper case in the row did not differ by Tukey's test ($P < 0.05$). Nd: not detected.

As reported in literature, the use of maltodextrin to encapsulate anthocyanins from blackberry pomace was efficient for the protection of phenolic compounds and anthocyanins in others conditions, as changes to light and temperature (Santos, Rodrigues, Costa, Bergamasco, & Madrona, 2017).

Evaluating the microencapsulation of astaxanthin of *Haematococ cuspluvialis* at 25 °C with maltodextrin and arabic gum applying spray dryer process, A study obtained half-life times between 1.6 and 2.5 days at different pHs (Bustos-Garza, Yáñez-Fernández, & Barragán-Huerta, 2013).

The addition of microencapsulated blackberry pomace is interesting in food products, some studies have used antioxidant products as a functional product. The type of food product and its sensory characteristics are important to choose the ideal product for application, such as yogurt and processed cheese (Batista et al., 2015; Belsito et al., 2017).

4. Conclusions

Extraction of antioxidant compounds from blackberry pomace is promising because it uses a by-product from the fruit processing industry. Maltodextrin microencapsulation of antioxidant compounds from this residue was efficient in reducing anthocyanin degradation against increasing pH. At lower pHs higher stability of the encapsulated samples was observed, the longer half-life and the lowest degradation constants were found. The microcapsules were efficient in reducing degradation of samples at lower pHs up to pH 5.0, it is possible to use them as a dye and as antioxidant compounds in several types of food, such as yogurt, dairy drinks, juices, sauces, soft drinks, jams, jellies, among others. The type of extraction is also important, the use of water in the extraction process was advantageous in relation to other solvents because of its safety and low cost.

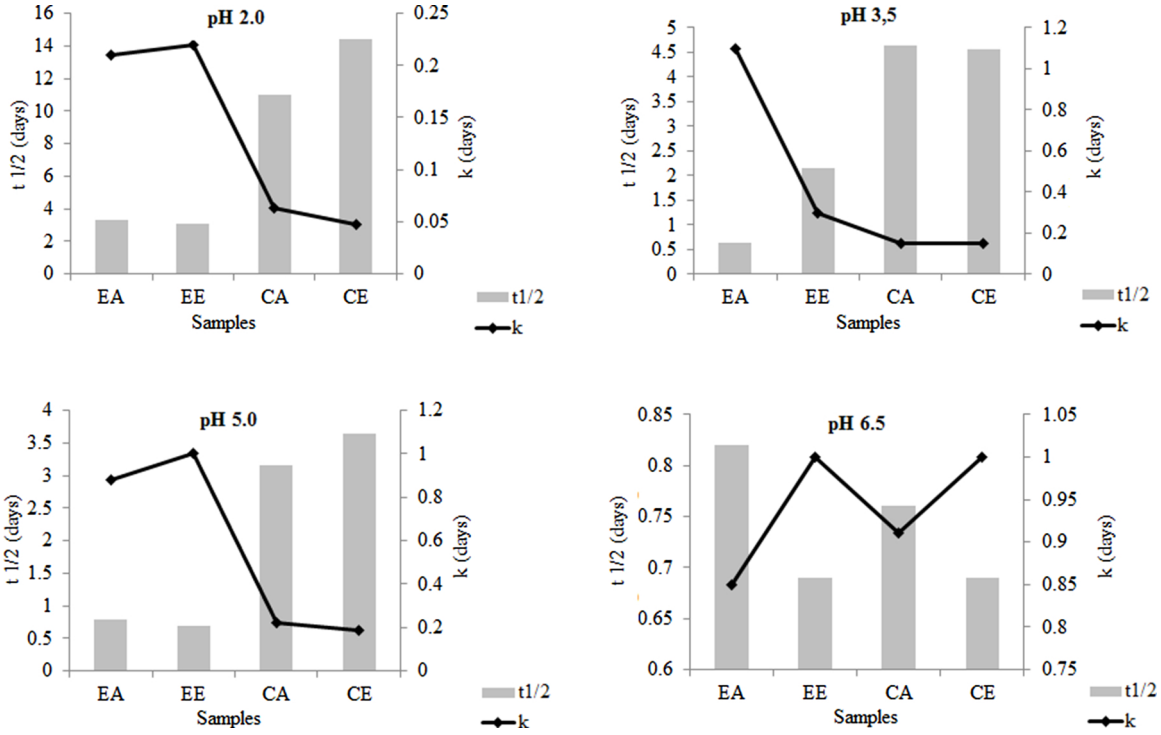

Fig. 2. Half-life ($t_{1/2}$) and sample degradation constant (k) EE, EA, CA and CE in different pHs.

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