Yellow passion fruit (*Passiflora edulis* f. *flavicarpa* Deg.) oil refining extracted by mechanical pressing

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Abstract— *Clarifiers clays are used in refinery operations of vegetable oils to remove pigments, improving their appearance, odor and flavor. In this study, yellow passion fruit (Passiflora edulis f. flavicarpa Deg.) seeds oil was extracted by mechanical pressing. Passion fruit seeds were dried in a convective dryer under temperatures of 50, 60 and 70°C in order to adjust different mathematical models to experimental data. Physico-chemical analyses were carried out of oil samples resulting from the refining process steps. Oil thermal performance was also evaluated during 90 days. Physico-chemical results varied in each refining stage: refractive index (1.4763 to 1.4666), water activity (0.9567 to 0.5140) and density (1.02 to 0.8914). Thermogravimetric (TGA), derivate thermogravimetric (DTGA) and differential scanning calorimetry (DSC) curves showed that refined oils presented a better thermal stability compared to the standard oil, without the clarification step.*

Keywords— clarifiers agents, oil extraction, oil refinery.

I. INTRODUCTION

Passion fruit belongs to the Passifloraceae family and is a native plant from tropical America, which comprises more than 500 species around the world. Among these, the yellow passion fruit (Passiflora edulis f. flavicarpa Degener) is widely cultivated and commercialized in Brazil, where it is presented in 95% of orchards [1,2,3].

Fruit juice and pulp industries produce thousands of tons of agro-industrial residues. During the processing of passion fruit juice, the main generated waste is composed of seeds and peels, which contains great amounts of fibres and oil. These type of raw material is applied in food, cosmetics and pharmaceutical industries as an alternative to reduce environmental impacts [4,5,6,7].

Food drying techniques result in mass and volume reductions as well as packaging and handling cost savings. They also minimize chemical and microbiological alterations during the final product storage [8]. Among various existing methods for drying, convective drying is one of the most used to remove moisture from a wide variety of biological materials, including grains and seeds [9]. This is due to the low cost of deployment, maintenance and ease of operation compared to other drying methods [10].

Passion fruit seeds present an excellent oil extraction percentage in the range between 18.5 and 28.3%, according to [3]. The oil also contains a huge amount of unsaturated fatty acids like oleic acid (C18:1), present in concentrations between 13.6 and 16.9%; and linoleic acid (C18:2), present in concentrations between 67.8 and 74.3%. These characteristics enable the use of passion fruit seed oil as a good source of food grade oils [3,11,12,13].

Crude vegetable oils, also named unrefined or unprocessed oils produced from vegetables, contain desirable compounds for human health like triacylglycerides (TAG), tocopherols (TCP) and phytosterols. However, they also present undesirable ones as free fat acids (FFA) and phospholipids [14]. The main purpose of oils extraction technologies is to separate proteins from fat obtaining a higher purity product using low cost processes which must avoid secondary chemical reactions [15,16].

In vegetable oils refinery industries, either by chemicals or physical procedures; blanching is a very important processing step where undesirable components are removed by the adsorption technique. Clays and adsorbents, like bentonite and activated carbon, are widely used once they present excellent adsorption characteristics and are able to remove phospholipids and minimize the increasing of free fat acids levels during blanching. Besides, the impurities removal allows the production of stable and clear oil; improves its sensory characteristics and oxidative stability [17, 18,19].

In the present study, we performed refinery of yellow passion fruit (*Passiflora edulis*) seeds oil extracted by mechanical pressing. The seeds were firstly dried and the best process conditions were chosen for the subsequent processing steps which were degumming, neutralization, clarification and filtration. The oil thermal performance was also analyzed using thermogravimetric (TGA) and differential scanning calorimetry (DSC) methods.

II. MATERIALS AND METHODS

2.1 Yellow passion fruit seeds drying

The experiment was conducted at Departamento de Tecnologia de Alimentos and Laboratório de Tecnologias Alternativas, both located at Universidade Federal de Sergipe (São Cristóvão *campus*).

Yellow passion fruit (*Passiflora edulis*) seeds with moisture content of 51.2 (kg water/kg dry matter) were obtained from a local juice industry specialized in tropical fruits. A sample size of 1.8 kg was used for study. The seeds were firstly placed in aluminum trays and set in a convective dryer at three different temperature conditions: 50, 60 and 70°C, based in articles of the kinetics of drying seed [7, 20, 21, 22]. The drying airflow temperature was monitored by a thermometer (INCOTERM, 5003, Brazil) installed inside the dryer. 600 g of seeds were used for each temperature. The moisture content was determined by gravimetric method where the set tray-sample was weighed in a semi analytical scale (METRA, M2202, Brazil) every 20 minutes until constant weigh, with three replicates [23]. The dried seeds were crushed in a Willey roller mill (TECNAL, TE-650, Brazil) and vacuum-packed in low-density polyethylene (LDPE) bags. Samples were stored at -18°C until oil extraction process.

2.2 Mathematical modeling of drying process

Yellow passion fruit seeds drying kinetics experimental data were adjusted according to the mathematical models of Brooker [24], Lewis [25], Fick [26] and residual [27] by nonlinear regression using the software STATISTICA 7.0®. The equations for each model are presented in Table 1.

TABLE 1
MATHEMATICAL MODELS USED TO ADJUST DRYING KINETICS EXPERIMENTAL DATA

Models	Expressions
Brooker [24]	$UR = c \times \exp(-k \times T)$
Lewis [25]	$UR = \exp(-k \times T)$
Fick [26]	$UR = a_1 \times \exp(-b_1 \times T) + a_2 \times \exp(-b_2 \times T) + a_3 \times \exp(-b_3 \times T)$
Residual [27]	$UR = a \times \exp(-b \times T) + c \times \exp(-d \times T)$

where:
a, b, c and d: models coefficients;
k: drying coefficient;
T: drying time.

The moisture content ratios and curves of yellow passion fruit seeds during drying were determined by the following equation (1) [27,28]:

$$UR = \frac{U - Ue}{Ui - Ue} \qquad \text{Equation (1)}$$

where:

UR: moisture content ratio, dimensionless;

U: moisture content (kg water/kg dry matter);

Ui: initial moisture content (kg water/kg dry matter);

Ue: balanced moisture content (kg water/kg dry matter).

2.3 Cold pressed extraction of yellow passion fruit seeds oil

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Yellow passion fruit seeds oil extraction was performed using a mechanical cold-press to obtain the crude oil, which was stored in an amber glass bottles at 10°C.

2.4 Refinery of yellow passion fruit seeds oil

Oil conventional refinery process consisted of four steps: (*i*) degumming; (*ii*) neutralization; (*iii*) clarification and (*iv*) filtration. During degumming, 3% of heated water was added to the oil volume in a stirring water bath used at 60°C during 30 minutes. Subsequently, the oil was centrifuged at 6000 rpm for 15 minutes for gum removal. For neutralization step, citric acid solutions (0.1 and 0.2% v/v) were added to the samples at 70°C for 10 minutes under continuous stirring. Yellow passion fruit seeds oil was neutralized by adding sodium hydroxide solution (2% v/v). Clarifiers clays (activated carbon and bentonite) were applied at clarification step in concentrations ranging from 1.0 to 5.0% each. Finally, the oil was poured onto filter paper. A vacuum pump (30 mmHg) was used to force the oil through the filter.

2.5 Physical analyses

Refraction index (327/IV) and density (337/IV) analyses were determined according to Instituto Adolfo Lutz [35]. Water activity was achieved using Aqualab equipment (Series 3, model TE, US).

2.6 Yellow passion fruit seeds oil thermal stability

Clarified refined oils (activated carbon and bentonite clarifier agents) and standard oil (none clarifier agent added) were analyzed by thermogravimetric (TGA), derivative thermogravimetric (DTGA) and differential scanning calorimetry (DSC) at the following storage periods: day zero, 45 days and 90 days.

TGA/DTGA and DSC curves were obtained through a thermobalance (TGA-60, SHIMADZU, Japan) and DSC-60 (SHIMADZU, Japan) cell under heating rate of 10°C.min^{-1}. The TGA/DTGA trials were performed with heating rate ranges from 25 to 900°C under N_2 dynamic atmosphere (100mL.min^{-1}). The DSC curves were obtained in a temperature ranging from 25 to 600°C under the same N_2 dynamic atmosphere [34].

III. RESULTS AND DISCUSSION

3.1 Drying kinetics and mathematical models adjustments

Passion fruit seeds drying curves presented decreasing periods due to the water activity reduction (Fig. 01). The best drying condition was at 70°C where the range time was shorter when compared to the other process conditions. Therefore, the increase in temperature reduced the drying time of the seeds [20]. Fig. 01 also presents a significant increasing in drying time even when different heating temperatures were applied. As yellow passion fruit is rich in soluble solids, these long drying periods might be related to the pulp residues presented around the seeds which could implicate in a superficial hardening [29]. In that case, there was a reduction on drying rates and final products presented a dryer surface however the interior was still moist as a result of surfaces modifications caused by high temperatures and formation of a hardy and impermeable peel

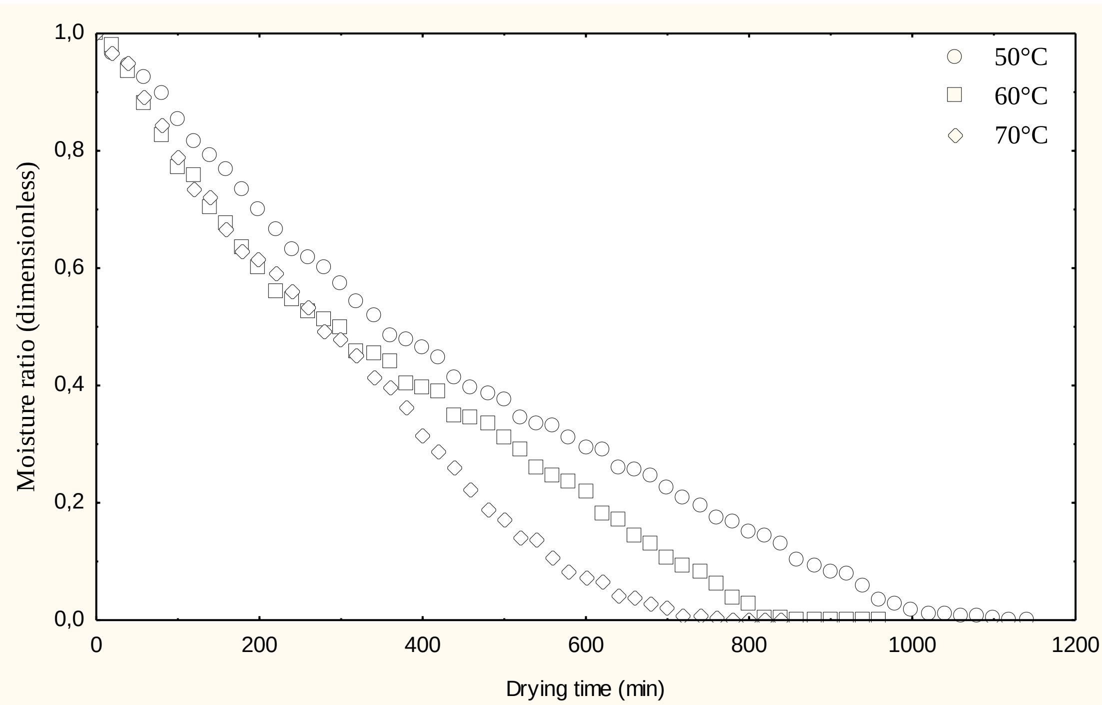

Fig 1. Moisture ratio curves during yellow passion fruit seeds drying at temperatures of 50, 60 and 70°C.

Table 2 shows models parameters values according to the independent variables (drying times) and dependent variables (moisture content), as well as mathematical models adjustments in different temperatures and respective correlation coefficients (R^2).

TABLE 2.
EQUATIONS PARAMETERS OF YELLOW PASSION FRUIT SEEDS DRYING KINETICS OBTAINED THROUGHOUT EXPERIMENTAL DATA

Parameters	Temperatures (°C)			Equation
	50	60	70	
C	1.0425	1.0590	1,1082	Brooker [24]
K	0.0019	0.0028	0,0034	
R^2	0.9884	0.9851	0,9814	
ε mean (%)	0.0094	0.0120	0,0155	
a_1	0.3602	0.3522	0.3691	Fick [26]
b_1	0.0023	0.0028	0.0034	
a_2	0.3602	0.3533	0.3691	
b_2	0.0023	0.0028	0.0034	
a_3	0.3602	0.3533	0.3692	
b_3	0.0023	0.0028	0.0034	
R^2	0.9884	0.9851	0.9814	
ε mean (%)	0.0897	0.0766	0.0995	
a	0.5403	0.5296	0.5535	Residual [27]
b	0.0023	0.0028	0.0034	
c	0.5403	0.5296	0.5535	
d	0.0023	0.0028	0.0034	
R^2	0.9884	0.9851	0.9814	
ε mean (%)	0.0724	0.1657	0.1689	
k	0.0021	0.0026	0.0030	Lewis [25]
R^2	0.9845	0.9831	0.9751	
ε mean (%)	0.0000	0.0000	0.0001	

It was found that for all drying temperature conditions, mathematical models adjusted to experimental data presented correlation coefficients (R^2) ranging from 0.9751 to 0.9884 (Table 2). The models of Brooker [24], Fick [26] and Residual [27] do not differ statistically among them for all three temperatures conditions (50, 60 and 70°C). However, Fick [26] and Residual [27] showed higher estimated mean errors. Among the models assessed, Brooker [24] and Lewis [25] presented the lowest standard deviation of estimate or estimated mean error (ε mean, %).

3.2 Physical analyses

Physical analyses of the yellow passion fruit seeds oil in each refinery process step are shown in Table 3. Refraction index analysis indicates a significant difference (significant level of 0.05) for all refinery steps. Nevertheless, there is no significant difference ($p \leq 0.05$) on refraction index among the clarified oils, which presented the lowest index values. Refraction index is related to the instauration degree of fatty acids, oxidative compounds and melting point [30].

Vegetable oils refraction index values ranges from 1.466 to 1.470 [34]. All the samples are in accordance with legislation, even when different clarification agents were used (Table 3). Results are similar to the ones reported by [1] and [11], whose passion fruit seeds oils presented refraction index of 1.4682 and 1.4691, respectively.

Water activity analyses results were significantly different ($p \leq 0.05$). Although, the oil clarified with bentonite agent presented the highest water activity.

Only the crude oil was significantly different ($p \leq 0.05$) from the oils resulting of refine process steps when density parameter was evaluated. That oil presented similar value to the density of fuels type A which standard density is 1.02 kg/L [36]. This high density value might be related to impurities which were present in the oil composition. Despite the oils derived from the refining process steps do not differ significantly from each other (5% significance level), the density values of degummed, neutralized and clarified oils with activated carbon agent, were above the limit allowed for marketing of refined vegetable oil, which is 0.926 g/cm³ [32].

TABLE 3.
PHYSICAL ANALYSES OF YELLOW PASSION FRUIT SEEDS OIL DURING REFINE PROCESS

Stage	Oil physical analyses		
	Refraction index	Water activity	Density
Crude oil	1.4763^a	0.9567^a	1.0261^a
Degummed oil	1.4733^b	0.5994^b	0.9345^b
Neutralized oil	1.4716^c	0.8974^a	0.9154^b
Clarified oil with activated carbon	1.4666^d	0.5140^b	0.9510^b
Clarified oil with bentonite	1.4668^d	0.8503^a	0.9126^b

Means with the same letter are not significantly different from each other ($p \leq 0.05$).

3.3 Comparative thermal analyses between the types of clarifiers in refine oil process

TGA, DTGA and DSC curve profiles of yellow passion fruit seeds oils were analyzed during the heating rate of 10°C.min^{-1} (Fig. 02). Thermal degradation curves of the standard oil (no clarifier) and refined ones (bentonite and activated carbon) show a similar behavior during the entire heating. This suggests that both standard and refined oils are thermally stable until the temperature of 330°C. However, the oil treated with bentonite begins its thermally degradation about 220°C. Despite that, all analyzed samples exhibited a single decomposition stage, which undergoes a complete degradation at 480°C. There was no residue at the end of the experiment.

The lower the reaction gap ($T-T_0$), the more stable is the material in terms of thermal decomposition. Therefore, refined oil clarified with activated carbon and the standard one (no clarifier) showed better thermal stability when compared to the bentonite treated oil at the starting point [33].

According to the thermogravimetric analysis carried out after 45 and 90 days, the curves indicate that the oils exhibit similar behavior indicating a single stage of decomposition. However, the refined oils show early decomposition at 330°C approximately, with complete degradation at temperature close to 480°C. Nonetheless, the standard oil began its decomposition at the temperature range from 260 to 300°C for the analysis of 45 and 90 days, respectively. Both ended at the temperature of 485°C, which means that the refined oils were more stable to decomposition. These oils also present higher oxidation temperature (OT) due to the formation of more stable compounds resulting on reduction of the levels of free fatty acids after the refining process [34].

DTGA curves (Fig. 02) showed similar decomposition stages at the initial condition, where a single endothermic event slowly occurs at temperatures close to 422°C and 428°C for clarified oil with activated carbon and standard, respectively; corresponding to water loss. DTGA curve for the oil clarified with bentonite presented two stages of decomposition. The first one occurs slowly with T_{DTGA} of 416°C and the second one around 468°C, proving the TGA curve, which also shows lower stability for the oil treated with bentonite.

After 45 days, DTGA curves present a similar behavior between samples of all refined oils, occurring single-stage decomposition which corresponds to an endothermic transition that starts at 410°C with a 52% mass loss for both samples that is related to water loss. For the standard oil sample, DTGA curve showed a single event which occurs slowly at a temperature range from 197.26 to 426.53°C, corresponding to an endothermic transition at 412°C with a mass loss of 241.30%. According to [31], in a study with buriti oil, the propagation of oxidation temperatures (OTonset) were lower for the crude oils, corroborating the greater thermo-oxidative fragility of crude oils when compared to the refined ones.

DTGA curves at 90 days appear to behave similarly, where a single event is related to degradation endothermic transition at temperatures around 415, 420 and 427°C for samples of oils treated with bentonite, activated carbon and standard.

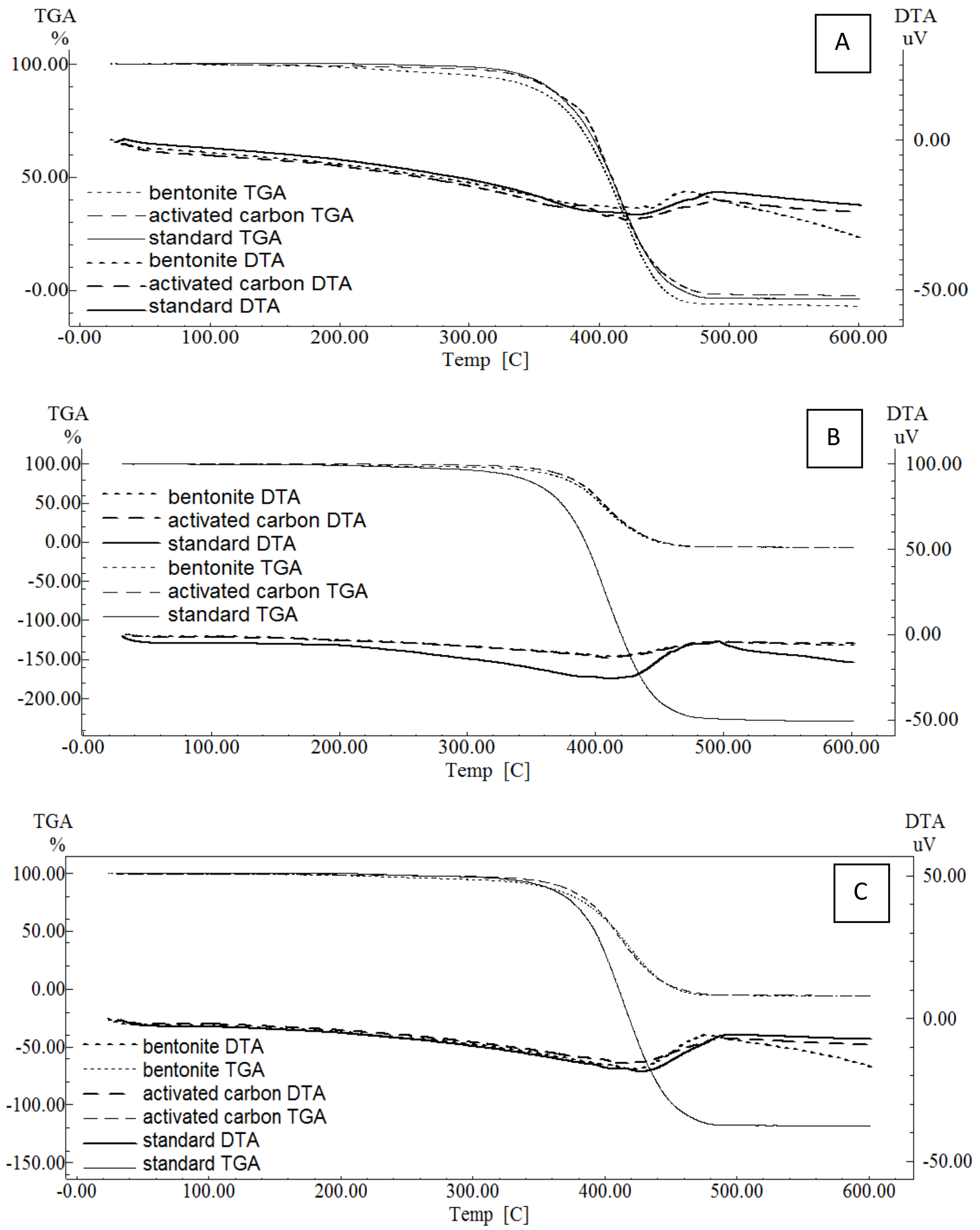

FIG 02. TGA/DTGA THERMAL ANALYSIS COMPARATIVE CURVES BETWEEN STANDARD AND REFINED OIL AT THE STORAGE PERIODS OF (A) DAY ZERO, (B) 45 DAYS AND (C) 90 DAYS

Through differential scanning calorimetry curve (DSC), it was possible to verify physical and chemical transitions that occur during the oil thermal decomposition. In the DSC curve (Fig. 03), the samples of standard oil and refined oils exhibit some similar behavior on the initial condition, presenting glass transitions and overlapping exothermic events. The first event exhibits a glass transition temperature (reversible event) that occurs slowly at 346.83°C for the standard oils and 360.55°C for the refined one with activated carbon. Refined oil with bentonite clay presented a single exothermic peak at 387.64°C that was attributed to residue thermal decomposition.

The curves for standard and activated carbon treated oil samples show that the second event is followed by a series of reactions which occur simultaneously under the main reaction at the temperatures range from 403.20°C to 407.00°C for the standard oil and activated carbon treated oil, respectively. The third event occurs only for the standard sample and shows an endothermic peak observed at 420.97°C, indicating no loss of water and volatiles compounds.

The DSC curve performed 45 days later, shows that the decomposition reactions occur between the temperature gap from 300°C to 443°C. The standard oil sample present three events, where the first reaction occurs slowly with an endothermic peak at 336.11°C representing loss of water and volatile reaction followed by two partially overlapped exothermic peaks corresponding to 363.99°C and 399.52°C, attributed to thermal decomposition of waste. The refined oils have similar DSC curves with two consecutive events. The first event represent an exothermic peak at 402.90°C and 403.93°C for the samples of bentonite and activated carbon treated oils, respectively. These results are related to the residue thermal decomposition, followed by endothermic reaction with peaks at 439.51°C and 427.85°C, probably due to loss of water and volatiles.

DSC analysis held at 90 days shows exothermic events. The first transition occurs slowly and in a discrete way between the samples at temperature about 355°C followed by a partially overlapping reaction at 384°C and an exothermic peak at approximately 477°C, relating to the main decomposition and irreversible thermal transition.

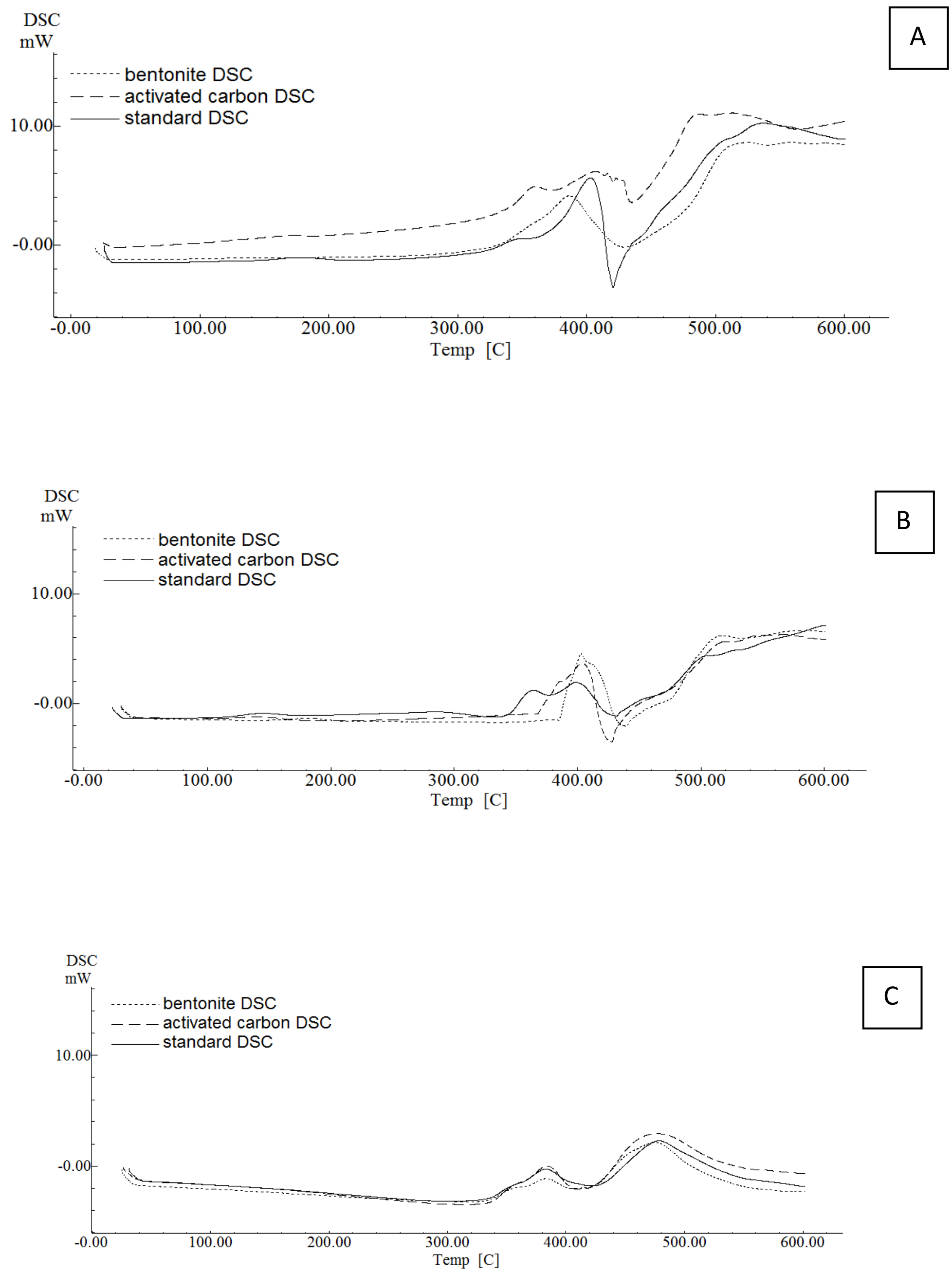

FIG. 3: DSC THERMAL ANALYSIS COMPARATIVE CURVES BETWEEN STANDARD AND REFINED OILS AT THE STORAGE PERIOD OF (A) DAY ZERO, (B) 45 DAYS AND (C) 90 DAYS

IV. CONCLUSION

Physico-chemical results varied in each refining stage performed: refractive index (1.4763 to 1.4666), water activity (0.9567 to 0.5140) and density (1.02 to 0.8914).

Among the mathematical models assessed to fit the experimental data of drying kinetics, Brooker and Lewis showed best adjustments as their determination coefficients were higher than 97% for all studied temperatures. They also presented low values of mean relative error (ε_{mean}, %).

Thermal analysis curves (TGA/DTGA and DSC) demonstrated that yellow passion fruit seed oils exhibit high thermal stability. However, the refined oils provide better thermal stability compared to the standard oil.

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