



Thermal study of chalcones

Thermal decomposition of chalcone and its hydroxylated derivatives

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Abstract

Chalcones α - β -unsaturated ketones are found in large plant species. Synthesis of chalcones and its three analogues hydroxy group at 2', 3' and 4' positions (**2–4**) was carried out. The studies of thermal behavior were made by thermogravimetry (TG) and differential scanning calorimetry, both under oxygen and nitrogen purge gases. In addition, the kinetic evaluation was carried out under heating rates of 5, 10 and 20 °C min⁻¹ with sample mass of 2 mg in open crucibles. The kinetic results obtained by TG analysis showed that the thermal behavior under oxygen shows that the functional hydroxy group substitution affects the thermal behavior of each molecule, with a gradual increase in the thermal decomposition. The activation energy (E_a /kJ mol⁻¹) showed under a nitrogen purge gas that the hydroxy group at 3' position (3'-hy-chalcone compound) has a different kinetic behavior, while the chalcone under oxygen showed a low activation energy when compared with the other hydroxy groups.

Keywords Chalcones · Thermal analysis · Kinetic behavior

Introduction

Chalcones are α - β -unsaturated ketones found in large plant species besides being a precursor for the biosynthetic route of flavonoids. Natural and synthetic chalcones have attracted attention in recent years due to the diversity of biological activities exhibited as anti-inflammatory [1], anticancer [2], antifungal [3] and leishmanicidal [4]. The synthetic preparation of chalcones involves Claisen–Schmidt condensation reaction between ketone and aldehyde

under acid or basic catalysis. Several other catalysis methods such as basic alumina, zinc chloride and Lewis acid such as BF₃ and AlCl₃ had been used [5].

The α - β -unsaturated bond conjugated to carbonyl acts as an electrophile and reacts with nucleophile allowing a cyclization via Michael's addition [6]. Structures containing hydroxy group in an *ortho* position might cyclize into flavanones and aurones. Thus, research studies the report of flavanones and aurones synthesis from chalcones and their biological activities [7, 8]. Due to their versatile structure, several synthetic routes were reported using chalcones as a precursor for getting heterocycles such as pyrazoline, oxiran, pyran, oxypyrimidine, isoxazoline [9], derivatives of pyridine [10], derivatives of benzheteroazepine [11]. Besides this, the chalcones or 1,3-diphenyl-2-propen-1-ones are considered as precursors of flavonoids and iso-flavonoids, which in turn affect the taste of foods, and they are also responsible for the color of flowers, fruits and leaves [12–15]. Furthermore, others interest in compounds **1–4** are correlated to their broad medicinal relevance, which includes antitumor (**1** and **2**) [16–18], antibacterial (**3**) [19], anti-inflammatory (**1**) [20] and antiprotozoal (**1** and **4**) [21, 22].

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Another application of these compounds relates to its in food technologies, which owing to the demand and interesting for new food antioxidants, it becomes very interesting to explore others molecules for the design, synthesis and characterization of new antioxidant agents in order to collaborate in food sciences [23–28]. Therefore, the aim of the present study was to describe thermal characterization of the chalcone (**1**) and its three analogues with hydroxy group at 2', 3' and 4' position (**2–4**), as seen in Fig. 1. The solid compounds were characterized by thermogravimetry (TG) and differential scanning calorimetry (DSC) at three heating rates and also under nitrogen and oxygen purge gases. In addition, TG curves were also used to obtain the kinetic information about the thermal decomposition stage.

Experimental

Synthesis of chalcones and their derivatives (Fig. 1: 1–4)

Chalcone (**1**) and its hydroxylated analogues (**2–4**) (Fig. 1) were synthesized by Claisen–Schmidt aldol condensation using protocols reported in previous studies from our group [29–31].

In a 30-mL vial, the appropriated acetophenone (2.5 mmol) and lithium hydroxide monohydrate (0.5 mmol) were dissolved in methanol (10 mL), and the mixture was stirred at 5 °C for 10 min. followed by the addition of benzaldehyde (2.70 mmol). The reaction mixture was stirred at room temperature and monitored by TLC using hexane/ethyl acetate (3:1) as the mobile phase. The reaction was quenched after 24 h by pouring into 100 mL of ice-cold water. In the synthesis of these compounds as seen in Fig. 1, from **1** to **4**, a stick mass was observed in the aqueous solution after quenching. Thus, the product was extracted by ethyl acetate (3 × 100 mL), dried over sodium sulfate and concentrated under reduced pressure. On the other hand, the synthesis of **2** and **3** led to precipitation after quenching with cold water, and it was then filtered and crystallized with hot acetone: ethanol (1:1). For compounds **1–4**, the crude products were purified by flash chromatography using hexanes: ethyl acetate as the solvent system in increasing order of polarity.

Characterization by nuclear magnetic resonance (NMR)

Chalcone (**1**) and its hydroxylated derivatives (**2–4**) were identified by ^1H and ^{13}C NMR spectra data obtained from Varian DRX-500 spectrometer (11.7 T). Chemical shifts (δ) were expressed in ppm. Coupling constants (J) were expressed in Hz, and splitting patterns are described as follows: s = singlet; br s = broad singlet, d = doublet; t = triplet; m = multiplet; dd = doublet of doublets. These data were extensively detailed in supporting information.

Characterization by thermal analysis

TG/DTG curves were obtained from a SDT 2960 model (TA Instruments). The evaluation of these compounds by TG analysis were carried out with sample masses around 2 mg in an α -alumina crucible with heating rates of 5, 10 and 20 °C min^{-1} under nitrogen and oxygen purge gases with flow of 100 mL min^{-1} . In addition, the kinetic study was performed using the three heating rates, which have been extensively used in previous studies [32–36]. Moreover, the use of this standard in kinetic analysis is very useful for future comparisons, besides allowing the reproducing of results obtained by us and also by other research groups. The kinetic methodology used in this work was proposed by Capela and Ribeiro, which is an isoconversional method and is based on approximation to the integral temperature on the convergent of a Jacobi fraction [37–40].

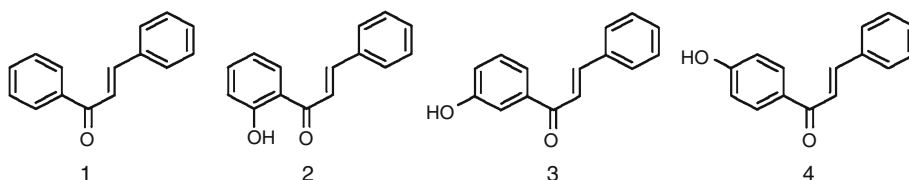
The DSC curves were carried out using a calorimeter SDT 2910 model, from TA Instruments, with heating rates of 20 °C min^{-1} from 30 to 400 °C under nitrogen and oxygen purge gases, with a flow of 50 mL min^{-1} .

Results and discussion

The TG/DTG evaluations of the chalcone and its hydroxylated derivatives under nitrogen and oxygen purge gases with mass of 2 mg and heating rate of 20 °C min^{-1} are shown in Fig. 2a–d, respectively.

The chalcone (Fig. 2a) shows that the initial thermal decomposition begins to take place after 105 °C going to 250 °C for both purge gases. Also it is possible to see that in oxygen, during the mass loss, there was a decrease in the

Fig. 1 Structures of chalcone (**1**) and its hydroxylated derivatives (**2–4**)



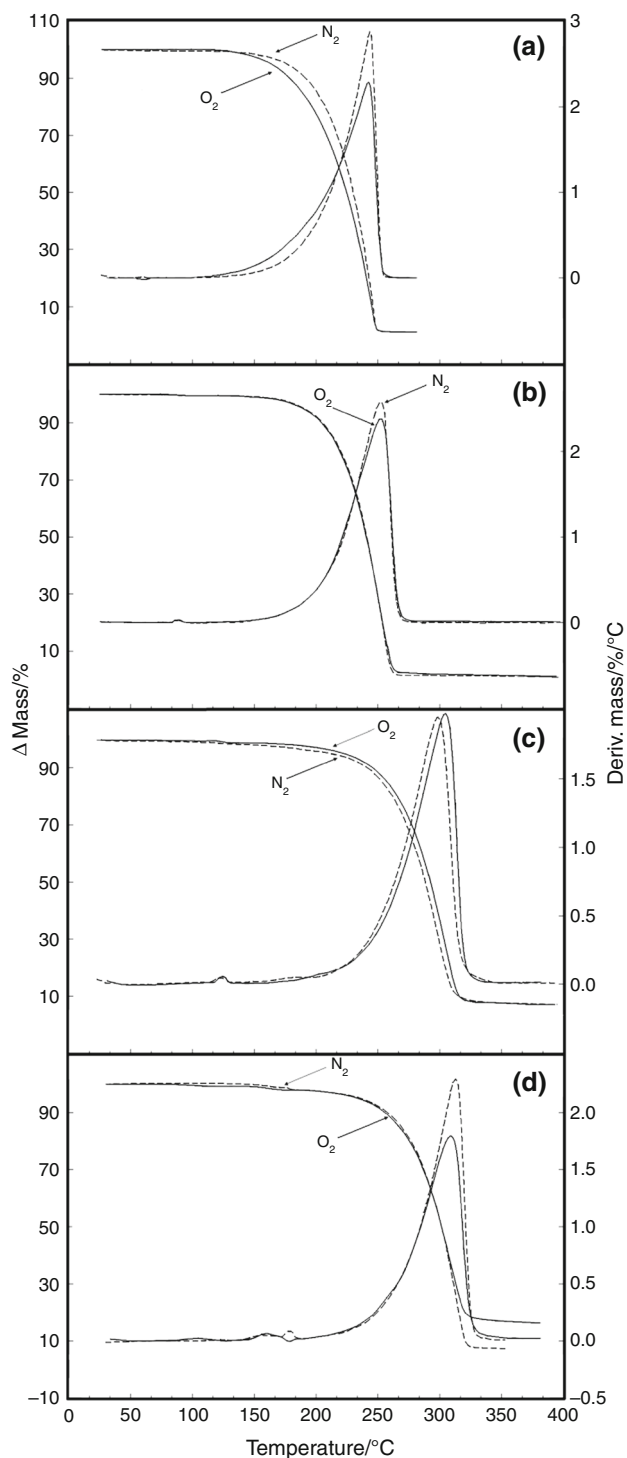


Fig. 2 TG/DTG curves of chalcone (a), 2'-hydroxychalcone (b), 3'-hydroxychalcone (c) and 4'-hydroxychalcone (d), with mass sample around 2 mg in α -alumina crucible, under oxygen and nitrogen purge gases and heating rate of $20\text{ }^{\circ}\text{C min}^{-1}$

temperatures, which is minor than that in nitrogen. The opposite effect under nitrogen gas can also be seen with 3'-hy-chalcone (Fig. 2c), where the reaction occurs at an interval of thermal decomposition from 150 to $326\text{ }^{\circ}\text{C}$.

These observed effects can be suggested as a relation of heat transfer and thermal conductivity of the gases with the samples.

The analyses with 2'-hy-chalcone (Fig. 2b) show that there is a similarity of thermal behavior of this sample under both purge gases. The DTG curve showed that the thermal decomposition stage occurs between 148 and $271\text{ }^{\circ}\text{C}$, without carbonaceous residues at the end of the reaction. For the analysis of 4'-hy-chalcone (Fig. 2d), it is possible to see that the thermal decomposition is also similar, but at the end of the reaction there was a difference in behavior. Other curves were made to verify this behavior, but the results were the same, being, therefore, as suggestion a possible reaction between the sample and the oxygen. For both analyses, there was the presence of carbonaceous residues, and the mass losses were 83.75 and 92.72% under oxygen and nitrogen purge gases.

For all samples seen in Fig. 2, it is possible to observe that there is an inflection in the TG/DTG curves between the initial temperature and before the thermal decomposition. This effect was attributed to the melting of the samples, which can be seen in the DSC curves in Figs. 3 and 4, which are associated with the same temperature as the TG/DTG curves. The DSC curves were carried out under nitrogen and oxygen purge gases, respectively, and the values of the melting points are indicated in the curves. The difference observed between the melting point analyses made with oxygen and nitrogen is attributed to the arrangement of samples in the crucible and also to the fact that samples analyzed are not a single crystal but are small different sized crystals.

For a better understanding and also for a comparison between these hydroxylated derivatives, the TG/DTG curves in oxygen and nitrogen purge gases were overlapping, as it is possible; see in Figs. 5 and 6, respectively.

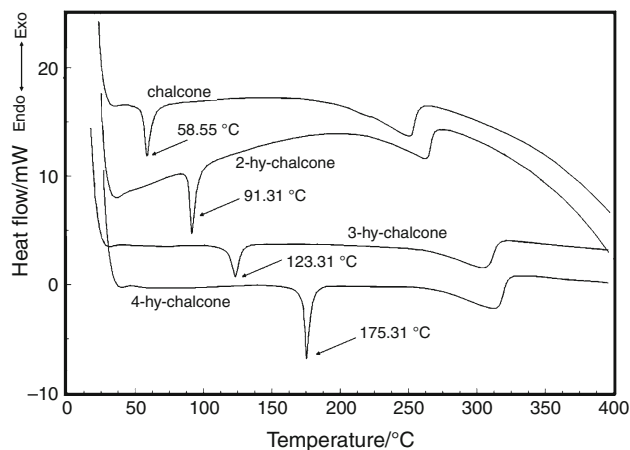


Fig. 3 DSC curves of chalcone and their hydroxylated derivatives compounds, with mass sample around 2 mg in aluminum crucible, under nitrogen purge gas and heating rate of $20\text{ }^{\circ}\text{C min}^{-1}$

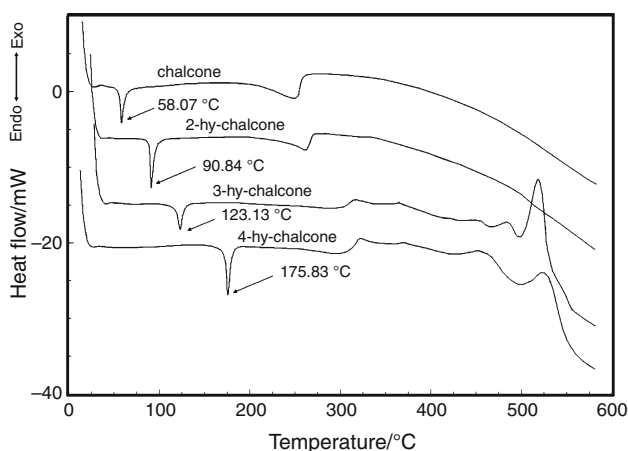


Fig. 4 DSC curves of chalcone and their hydroxylated derivatives compounds, with mass sample around 2 mg in aluminum crucible, under oxygen purge gas and heating rate of $20\text{ }^{\circ}\text{C min}^{-1}$

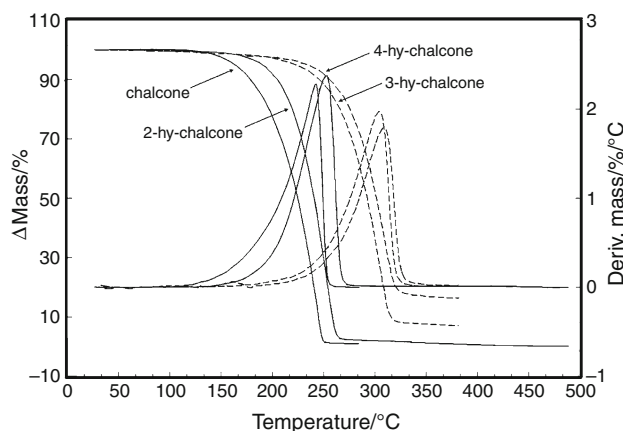


Fig. 5 TG/DTG curves of chalcone and their hydroxylated derivatives compounds, with mass sample of 2 mg in α -alumina crucible, under oxygen purge gas and a heating rate of $20\text{ }^{\circ}\text{C min}^{-1}$

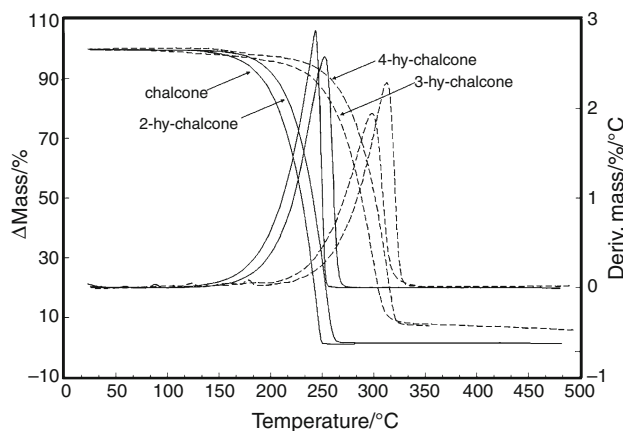


Fig. 6 TG/DTG curves chalcone and their hydroxylated derivatives compounds, with mass sample of 2 mg in α -alumina crucible, under nitrogen purge gas and a heating rate of $20\text{ }^{\circ}\text{C min}^{-1}$

The thermal behavior in oxygen shows that the functional hydroxy group substitution present at each position in the chalcone affects the thermal conductivity of the molecule, because there was a gradual increase in the thermal decomposition temperature. In addition, the thermal decomposition at 3' and 4'-hy-chalcone shows a difference in carbonaceous residues generated at the end of the reaction. However, this fact is not seen for the chalcone and 2'-hy-chalcone. For nitrogen analysis, the thermal behavior is similar to that seen in oxygen purge gas, except that there were no carbonaceous residues generated at the end of the thermal decomposition.

Kinetic evaluation

Figure 7 shows the overlapping of the three TG curves with heating rates of 5, 10 and $20\text{ }^{\circ}\text{C min}^{-1}$ of chalcone as a set of curves to obtain the kinetic data. The other compounds were not placed because they follow the same tendency of displacement of the heating rates. Moreover, in others works, we have used a pattern of at least three TG curves for kinetic behavior analysis, as suggested by the ICTAC committee [40–45]. In a similar way, for all curves (even for those not shown), the result shows that there were no changes to the thermal behavior, and therefore the profiles of curves remained the same, that is, without apparent overlapping of thermal decomposition reactions.

Table 1 shows the average of the values of activation energy (with a coefficient variation), the linear correlation obtained for compounds and the temperature intervals in DTG curves that were used for kinetic evaluation. The low activation energy values obtained for these compounds indicate the dependence on temperature, which implies that low temperatures are required for thermal decomposition.

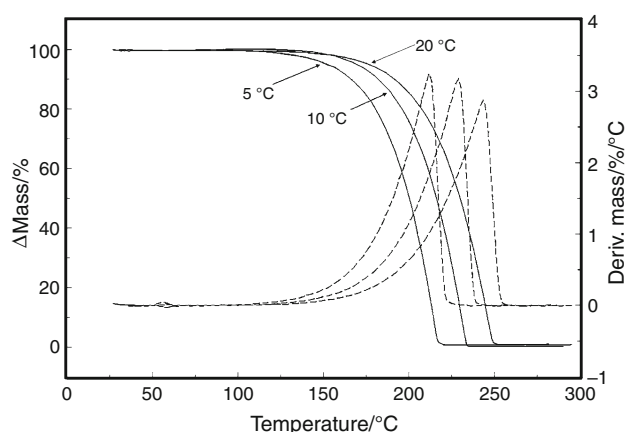


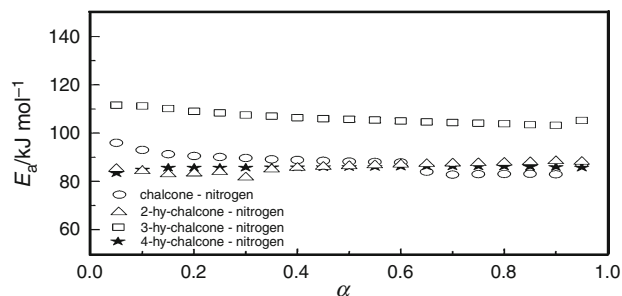
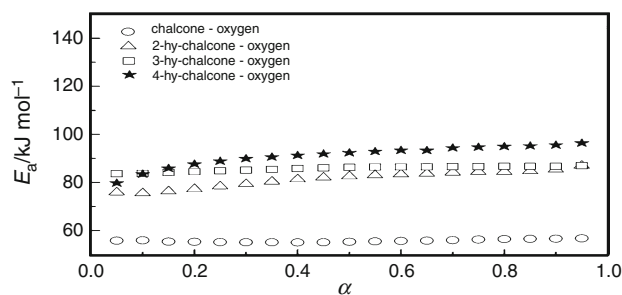
Fig. 7 TG/DTG curves of chalcone with sample mass around 2 mg in α -alumina crucible, under nitrogen purge gas and a heating rates of 5, 10 and $20\text{ }^{\circ}\text{C min}^{-1}$

Table 1 E_a (kJ mol^{-1}), correlation coefficient (r) and temperature intervals of DTG curves for thermal decomposition

Compounds	Purge gases	Temperature ranges (DTG curves)	$*E_a/\text{kJ mol}^{-1}$	$*r$
Chalcone	Nitrogen	(5 °C) 121–225 °C	87.76 ± 0.04	0.99646
		(10 °C) 136–236 °C		
		(20 °C) 148–257 °C		
Chalcone	Oxygen	(5 °C) 105–219 °C	55.72 ± 0.01	0.96343
		(10 °C) 114–230 °C		
		(20 °C) 121–255 °C		
2'-Hydroxychalcone	Nitrogen	(5 °C) 137–243 °C	85.97 ± 0.02	0.99661
		(10 °C) 152–256 °C		
		(20 °C) 162–272 °C		
2'-Hydroxychalcone	Oxygen	(5 °C) 122–237 °C	81.56 ± 0.04	0.99646
		(10 °C) 150–262 °C		
		(20 °C) 168–272 °C		
3'-Hydroxychalcone	Nitrogen	(5 °C) 181–290 °C	106.45 ± 0.02	0.99964
		(10 °C) 200–314 °C		
		(20 °C) 211–330 °C		
3'-Hydroxychalcone	Oxygen	(5 °C) 168–290 °C	85.76 ± 0.01	0.99694
		(10 °C) 187–309 °C		
		(20 °C) 201–329 °C		
4'-Hydroxychalcone	Nitrogen	(5 °C) 186–299 °C	85.77 ± 0.01	0.99895
		(10 °C) 200–319 °C		
		(20 °C) 221–337 °C		
4'-Hydroxychalcone	Oxygen	(5 °C) 194–302 °C	91.20 ± 0.04	0.99547
		(10 °C) 203–315 °C		
		(20 °C) 218–334 °C		

*Average

The relation between the activation energy versus conversion degree under nitrogen and oxygen purge gases is shown in Figs. 8 and 9, respectively. As can be seen in the nitrogen purge gas, the kinetic behaviors are linear, with a more pronounced difference for 3'-hydroxychalcone, while for analysis under oxygen purge gas, the major difference occurs for chalcone. It is important to note that the kinetic behavior in oxygen follows a pattern of minor activation energy for chalcone to major activation energy for 4'-hy-chalcone. The minor activation energy of chalcone under oxygen is probably due to the weak intermolecular

**Fig. 8** Calculated $E_a/\text{kJ mol}^{-1}$ as a function of α for the transition phase stage under nitrogen purge gas**Fig. 9** Calculated $E_a/\text{kJ mol}^{-1}$ as a function of α for the transition phase stage under oxygen purge gas

interactions, while for the other molecules this interaction is similar, hence the motive of the closest activation energies. However, under nitrogen, the 3'-hy-chalcone has major value of activation energy, which stands out from the others. This behavior is a probable indication that this molecule has an intermolecular interaction greater than the others. In previous papers about the synthesis and thermal characterization of flavanone and 6'-hydroxyflavanone flavanones, it was possible to see that there was an alteration in the activation energy under nitrogen purge gas with

hydroxy group in flavanone, with the increase in the activation energy to a higher value [26].

Conclusions

In the present study, we evaluated four chalcones by thermogravimetry (TG), derivative thermogravimetry (DTG) and differential scanning calorimetry (DSC), which showed the comparisons of thermal behavior under nitrogen and oxygen purge gases. Besides, the present study on these molecules showed that they have a thermal behavior without presenting large variations of overlapping reactions, which can be used as analysis standards. The thermal decomposition showed that these compounds had homogeneous processes, and therefore they provide a good and linear behavior for kinetic study. The kinetic evaluation showed that the third position of the hydroxy group at aromatic ring affects the kinetic behavior, as seen in the nitrogen purge gas, but the analysis under the oxygen indicates that the position little affects the kinetic behavior.

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