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Thermal studies of *Albizia inopinata* crude extract in the presence of cyclodextrin and Aerosil® by TG and DSC coupled to the photovisual system

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Abstract

Albizia inopinata (Harms) G.P. LEWIS has been studied for its anti-hypertensive/vasodilater phytopharmaceutical effects. In this work TG and DSC are coupled to a photovisual system and were used to determine the thermal behavior of *A. inopinata* dried extract prepared with and without stabilizers. The extracts were prepared by percolation of the powdered leaves in an ethanol:water (70:30) solvent system for 72 h and then dried using a spray–dryer technique. Aerosil® 200 (Colloidal silica dioxide) and β-cyclodextrin were used as stabilizers for the preparation of the dried extracts. TG curves were obtained using a Shimadzu thermobalance, model TGA-50H. DSC curves were obtained with a Shimadzu calorimeter, model DSC-50, coupled to the photovisual system. TG curves for *A. inopinata* extracts and Albizia-stabilizers exhibited six thermal decomposition stages. DSC curves showed phase transition differences among the samples. The pictures obtained by DSC photovisual showed thermal behavior differences between the products.

Keywords: *Albizia inopinata*; Adjuvants; Thermal analysis

1. Introduction

In the pharmaceutical industry the use of suitable technological adjuvants along with spray–drying technology represents an important step in the assurance of the adequate stability and quality of plant extracts. This is due to the fact that adjuvants can be changed and consequently influence the bioavailability of the products.

Albizia inopinata (Harms) G. P. LEWIS (*Leguminosae*), is a plant used mainly for ornamental purpose. It is popularly known as ‘‘bordão de velho’’ and ‘‘casqueiro’’. In Brazil, the species *A. inopinata* is sparsely distributed in the country, and is especially abundant in the northeast. A computer-aided search (NAPRALERT, College of Pharmacy, University of Illinois at Chicago), covering the period of 1975–1998, showed ethnopharmacological references to the use of the bark of *A. julibrissin* for the treatment of insomnia [9], and the bark of *A. lebbek* for the treatment of asthma in India [7]. There is also a report on the use of *A. zigia* in psychiatric disorders in Nigeria [1]. It has been reported that *A. chinensis*, *A. grandibracteata* and *A. gummifera* showed hypotensive activity in cats, guinea-pigs, monkeys and rabbits [4]. Whereas, *A. lebbek* and *A. lebbekioides* were active in dogs [2,5]. Finally, Dhawan et al. [3]

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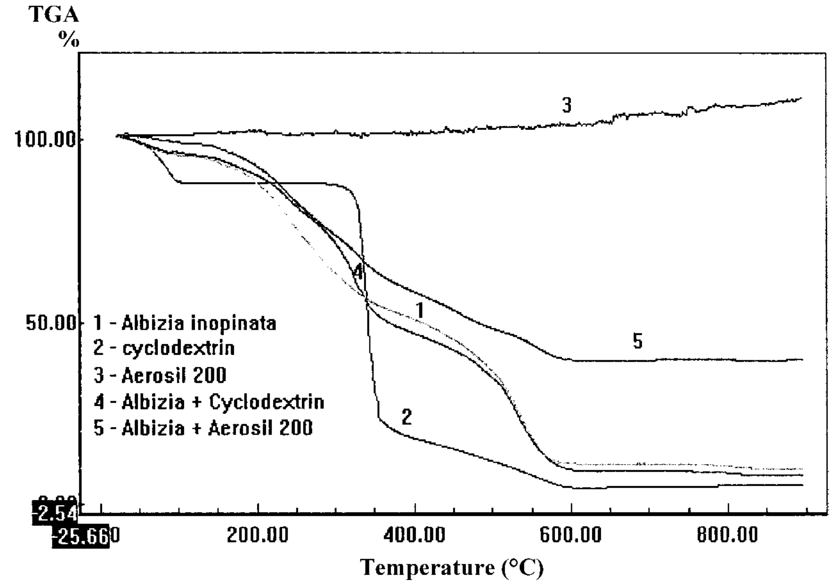

Fig. 1. TG of *A. inopinata* without and with stabilizers.

and Vohra et al. [8] reported a diuretic activity of *A. julibrissin* and *A. odoratissima* in rats.

In a recent paper, we demonstrated that the ethanol extract of the leaves of *A. inopinata* presented a marked hypotensive effect. This is due to a peripheral vasodilation, at least partly secondary to the release of nitric oxide by the vascular endothelium [6], thus indicating that this extract could be used as an anti-hypertensive/vasodilater agent.

In this work TG and DSC data were used to determine the thermal behavior of the *A. inopinnata* dried extract with or without stabilizers.

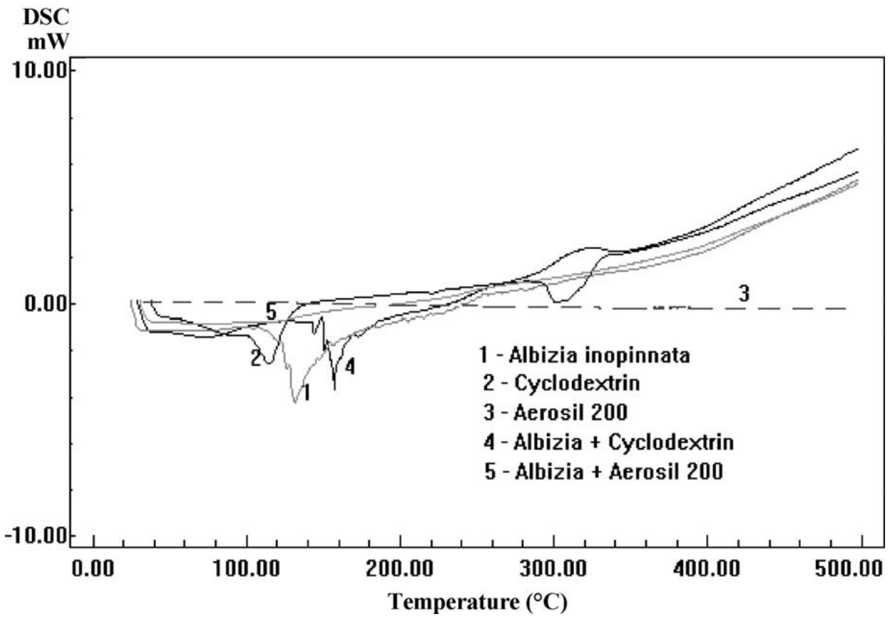

Fig. 2. DSC of *A. inopinata* without and with adjuvants and adjuvants alone.

Table 1
Thermal decomposition constant (K) of the *A. inopinata* extracts

Temperature (°C)	K		
	A. inopinata extract	*A. inopinata* + cyclodextrin	*A. inopinata* + Aerosil® 200
120	–	2.20×10^{-5}	2.29×10^{-5}
130	2.29×10^{-5}	2.94×10^{-5}	2.44×10^{-5}
140	3.28×10^{-5}	3.48×10^{-5}	7.55×10^{-5}
150	8.78×10^{-5}	4.71×10^{-5}	8.71×10^{-5}

2. Experimental

2.1. Preparation of the aqueous fraction of the leaves (AFL) of A. inopinata (Harms) G.P. LEWIS

Mature leaves of *A. inopinata* (Harms) G.P. LEWIS were collected in April 1995 from the district of Santa Rita, Paraíba (voucher specimen Code Agra and Góis 3599) and were dried at 40 °C in an oven and pulverized. The extract was obtained by percolation of powdered leaves in the ethanol:water (70:30) solvent system for 72 h and then dried by spray–drier Labplant, model SD-05. Colloidal silica dioxide (Aerosil® 200) and β-cyclodextrin (Kleptose®) were used as stabilizers for the preparation of the dried extracts.

2.2. Thermal analysis

The TG curves were obtained on a Shimadzu thermobalance model TGA-50H under air atmosphere

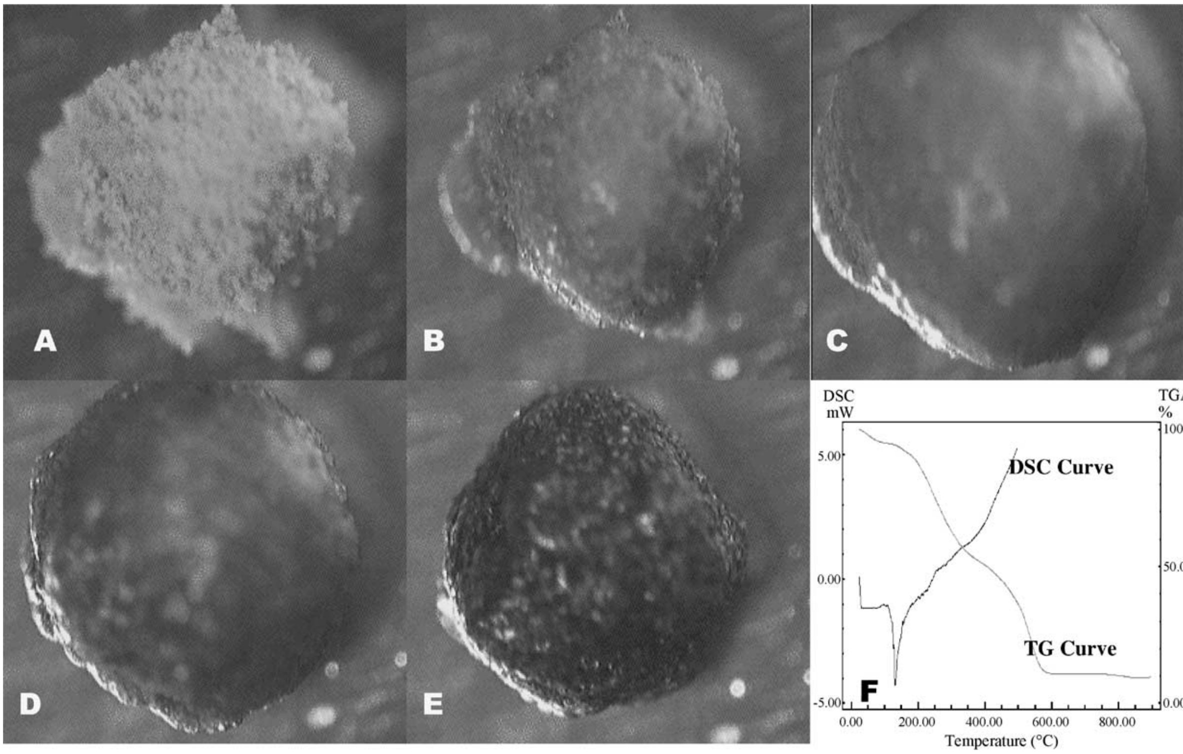

Fig. 3. DSC photovisual of the *A. inopinata* without adjuvants: (A) 29 °C; (B) 155 °C; (C) 170 °C; (D) 205 °C; (E) 230 °C; (F) TG and DSC curves.

with a 20 ml/min flow rate, using a heating rate of 10 °C/min up to 900 °C. The samples were packed in an alumina cell with a mass of around 10 mg.

The DSC curves were obtained using a Shimadzu calorimeter, model DSC-50, coupled to a photovisual system, oven with a temperature range of 25–500 °C, under an atmosphere of nitrogen with constant flow of 50 ml/min and a heating rate of 5 °C/min. The samples were packed in aluminum cells. The mass used was approximately 2 mg.

The TG isothermal curves were measured at the following temperatures: 120, 130, 140, and 150 °C for 240 min. The rate constants for the thermal decomposition reactions were determined from thermogravimetric data using the Arrhenius expressions. The curves were analyzed with Shimadzu TASYS software.

3. Results

Fig. 1 shows the TG curves for *A. inopinata* extracts without and with the stabilizers cyclodextrin and aerosil.

Fig. 2 represents the DSC curves for *A. inopinata* extracts without and with the stabilizers cyclodextrin and aerosil.

The rate constant data showed that the thermal decomposition reactions for *A. inopinata* extract are different as seen in Table 1. The rate constant values were obtained using the Arrhenius classical equations.

The DSC and TG curves and pictures for the dried extract of *A. inopinata* without adjuvant are shown in Fig. 3.

The Figs. 4 and 5 show the TG/DSC curves and pictures for the dried extracts of *A. inopinata* with stabilizers cyclodextrin and aerosil, respectively.

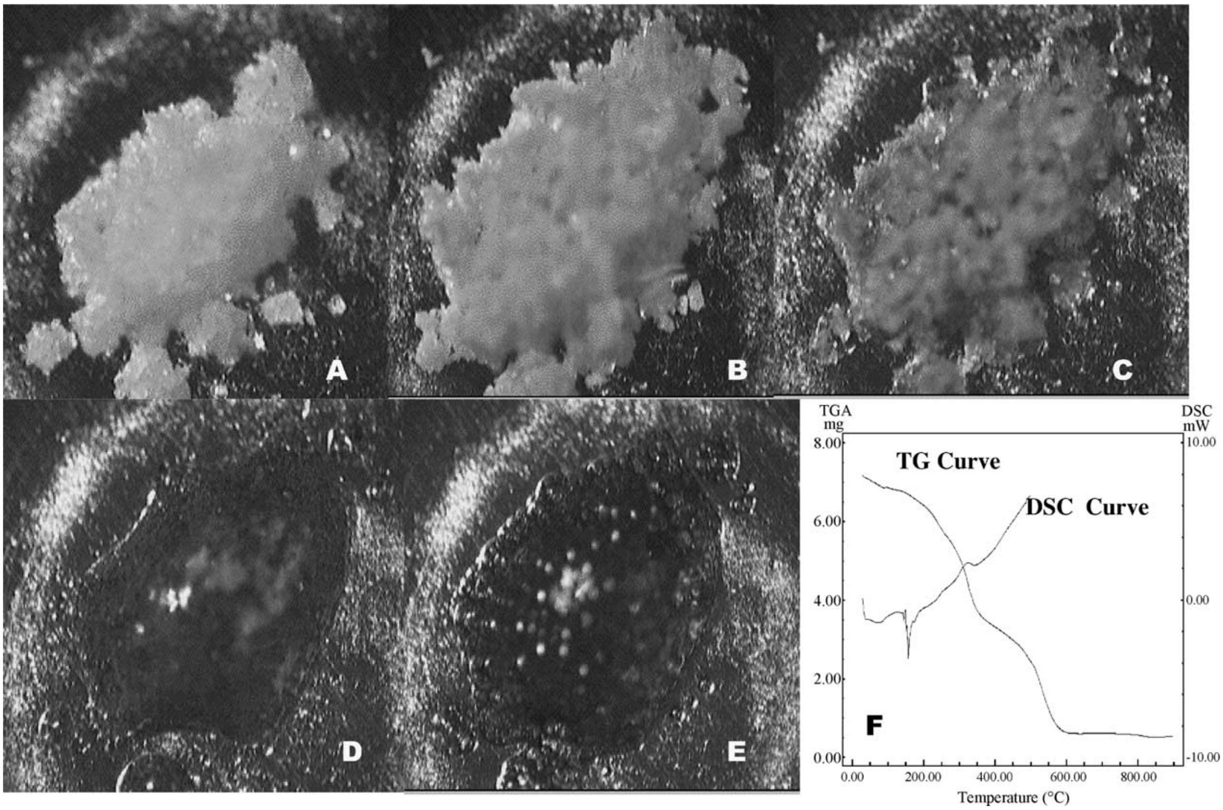

Fig. 4. DSC photovisual of the *A. inopinata* with β-cyclodextrin: (A) 25 °C; (B) 237 °C; (C) 241 °C; (D) 248 °C; (E) 280 °C; (F) TG and DSC curves.

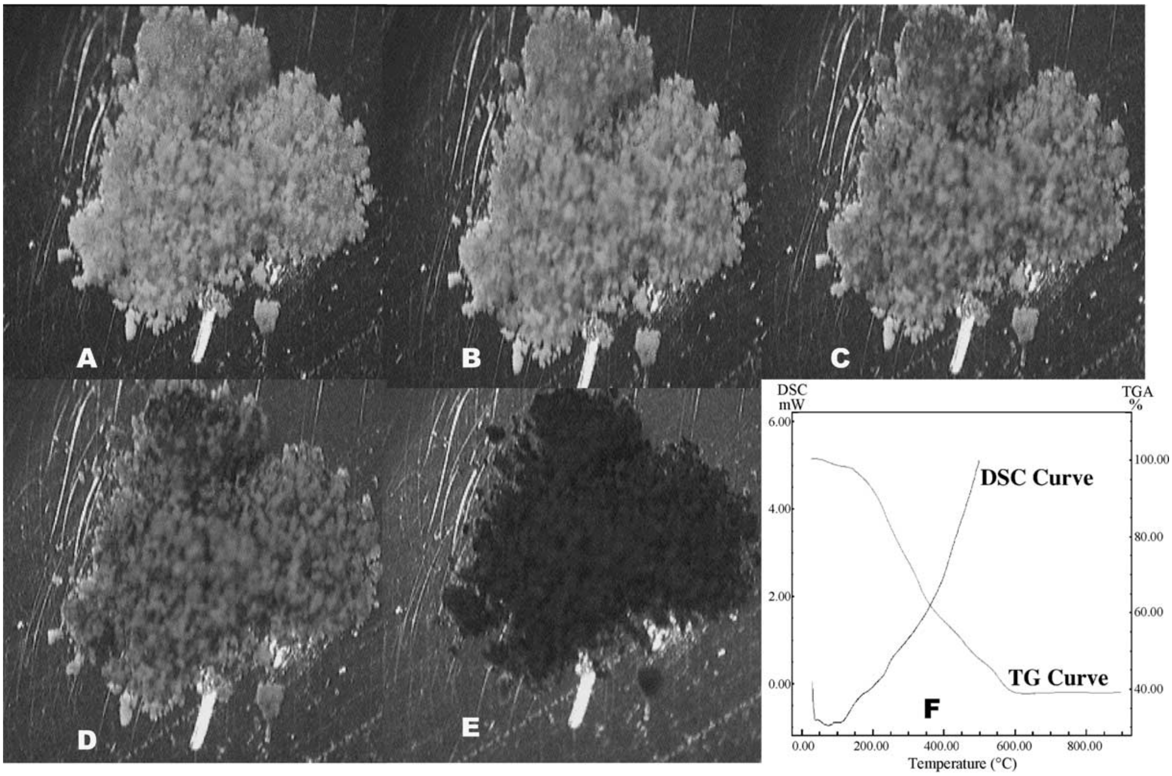

Fig. 5. DSC photovisual of the *A. inopinata* with Aerosil® 200: (A) 27 °C; (B) 135 °C; (C) 175 °C; (D) 248 °C; (E) 330 °C; (F) TG and DSC curves.

4. Discussion

The TG curves for the *A. inopinata* (Fig. 1) without adjuvant, analyzed by the tangent method, showed seven thermal decomposition processes, and had a mineral residue of 10.32%. The spray–dried extract with β-cyclodextrin showed eight thermal decomposition processes, with a mineral residue of 8.56%. The spray–dried extract with colloidal silica dioxide showed seven processes, with a mineral residue of 38.58%. The thermogravimetric data allowed for the calculation of kinetic parameters in a temperature range of 120–150 °C.

The rate constants for the thermal decomposition reaction revealed the best stability for dried extract *A. inopinata* containing cyclodextrin like stabilizer. The thermal decomposition reaction obeyed second-order kinetics.

The DSC curve for the dried extract of *A. inopinata* without adjuvant presented an endothermic peak at 131.84 °C and a heat in 162.18 kJ/kg (Fig. 2). Its decomposition happened with turbulence formation, involving processes of product vaporization which was evidenced by the DSC photovisual process. In pictures A–C (Fig. 3), a thermal behavior with fast decomposition of product can be observed.

The dried extract with β-cyclodextrin showed a peak at 157.22 °C and a heat at 47.14 kJ/kg. The DSC curve (Fig. 2) showed that cyclodextrin displaced the endothermic peak to a higher temperature in relation to the dried extract of *A. inopinata* without stabilizers (Fig. 2). Pictures A–C (Fig. 4) confirm the best stability of the product when compared with the dried extract of *A. inopinata* without stabilizer.

The dried extract of *A. inopinata* with colloidal silica dioxide presented endothermic peak at temperatures 40.31, 74.78 and 105.53 °C followed by exothermic process of thermal decomposition (Fig. 2). The pictures A–D and F (Fig. 5) showed a dried product with good appearance, but its thermal behavior evidenced smaller stability than the other products.

5. Conclusion

The rate constants of the thermal decomposition reaction and DSC photovisual pictures of the different extracts of the *A. inopinata* revealed that the stabilizer cyclodextrin produced the larger stable extract.

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