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Effect of pH and temperature on tropane alkaloids within a processing strategy to provide safe infant cereal-based food

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ABSTRACT

Tropane alkaloids (TAs) are secondary metabolites from weeds that can contaminate cereals and vegetables during harvest. Due to their toxicity, the Regulation (EC) 2023/915 sets maximum levels for atropine and scopolamine in cereal-based foods for infants containing millet, sorghum, buckwheat or their derived products. The aim of this study was to evaluate the effect of pH and temperature on the stability of TAs, as possible parameters in thermal processing to mitigate this chemical hazard in cereal-based infant food. The effect of pH (4 and 7) and temperature (80 °C and 100 °C) was assessed in buffer solutions. Also, treatment at 180 °C was performed in spiked and naturally incurred millet flour to assess the effect of high temperature, simulating cooking or drying, on the stability of TAs in the cereal matrix. The fate of 24 TAs was assessed by UHPLC-MS/MS. TAs showed high thermostability, although it was variable depending on the specific compound, pH, temperature and treatment time. In buffer solutions, higher degradation was found at 100 °C and pH 7. In spiked millet flour at 180 °C for 10 min, scopolamine and atropine contents decreased by 25 % and 22 %, similarly to other TAs which also showed a slow thermal degradation. Atropine, scopolamine, anisodamine, norscopolamine, scopine and scopoline were found in naturally contaminated millet flour. Interestingly, naturally incurred atropine was more thermostable than when spiked, showing a protective effect of the cereal matrix on TAs degradation. The present results highlight the need for an accurate monitorization of TAs in raw materials, as this chemical hazard may remain in infant cereal-based food even after intense thermal processing.

1. Introduction

Cereal-based infant foods are an important source of energy, protein, vitamins and minerals for infants from 6 to nearly 36 months old to avoid malnutrition (European Parliament, 2013; Roess, Jacquier, Catellier, Carvalho, Lutes, Anater, & Dietz, 2018). Cereal-based infant foods can be made with several species of cereals such as millet, barley, corn, oats, quinoa, rice, rye, sorghum, triticale and wheat (Jeelani et al., 2020). These cereals can be contaminated with seeds and other plant parts that contain tropane alkaloids (TAs). TAs are secondary metabolites which naturally occur in plants of several families including Brassicaceae, Convolvulaceae, Solanaceae (e.g., mandrake, henbane, deadly nightshade, Jimson weed) and Erythroxylaceae (EFSA CONTAM Panel, 2013). More than 200 different TAs have been identified and can be found in every part of a plant, including seeds, fruits, flowers leaves and stems, so cross-contamination is frequent due to fast and mechanical harvesting (EFSA CONTAM Panel, 2013; Kohnen-Johannsen & Kayser, 2019). Among all known families of TAs, EFSA CONTAM Panel (2013) concluded that Datura Tas can be considered among the most relevant for the food safety of consumers. TAs are primarily antagonists of the muscarinic acetylcholine receptors. In humans, the predominant peripheral antimuscarinic effects are decreased production of salivary secretions; bronchial and sweat glands; dilation of the pupils (mydriasis); paralysis of accommodation of the eye; change in heart rate; inhibition of micturition; reduction in gastrointestinal tone and inhibition of gastric acid secretion (Mulder et al., 2016). Datura TAs can be divided in three subgroups: *i)* low molecular weight TAs (LMW), which contain only the tropane ring; *ii)* Convolvulaceae-type TAs, which contain a tropane ring esterified to a benzoic acid derivative and are found in the family of Convolvulaceae; and *iii)* Datura-type TAs, which have a

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tropane ring esterified to a phenylacetic acid derivative (Mulder et al., 2016).

Due to the toxic effects, the European Commission set legal limits for Datura-type TAs in baby foods and processed cereal-based foods for infants and young children containing millet, sorghum, buckwheat or derived products (European Commission, 2016). The maximum level permitted in these products are 1 μg/kg for atropine and scopolamine (European Commission, 2023). Besides the most studied TAs, atropine and scopolamine, other TAs can be present in food (Boros et al., 2010; Caligiani, Palla, Bonzanini, Bianchi, & Bruni, 2011; Chen, Marín-Sáez, Romero-González, & Garrido Frenich, 2017; González-Gómez, Morante-Zarcero, Pérez-Quintanilla, & Sierra, 2022). Particularly, the presence of atropine and scopolamine above these limits has been documented in cereal-based foods for infants and children (Torrents-Masoliver et al., 2022). An average of 4.6, 4.4 and 0.5 μg/kg of TAs in 2011, 2012 and 2014 was found in cereal-based food for infants and young children, with maximum levels of 80.8, 57.6 and 3.9 μg/kg, respectively, in the Netherlands (Mulder, Pereboom-de Fauw, Hoogenboom, de Stoppelaar, & de Nijs, 2015). Mulder et al. (2016) analysed 260 samples of cereal-based foods for children under 36 months, where pasta and cereal-based meals (18 samples) were the most contaminated (55.6 % of positive samples above LOD) with an average concentration of 131 μg/kg and a maximum concentration of 860 μg/kg. Millet flour was highlighted for its high incidence (23.5 %) and levels (upt ot 361.2 μg/kg) of TAs (Mulder et al., 2016). Lower incidence (13.1 % and 14.6 %, respectively) was found in cookies for children and biscuits and pastry, with maximum concentrations of 86 μg/kg and 12 μg/kg, respectively. Another study found 11.5 μg/kg of atropine and 2.8 μg/kg of scopolamine in cereal-based baby food (Marín-Sáez, Romero-González, & Garrido Frenich, 2019).

Food safety alerts have been recently issued in Rapid Alert System for Food and Feed (RASFF, notification reference *2023.3153; 2023.4419*) regarding the detection of atropine and scopolamine far above the regulated limit in teff flour in Spain (130.7 μg/kg and 41.8 μg/kg, respectively for atropine and scopolamine) (May 2023, ref. *2023.3153*) and gluten-free chocolate cooked made with teff flour (July 2023, ref. *2023.4419*).

Common cleaning practices during cereal processing are not always sufficient to remove the weed plant parts responsible for the TAs presence (Adamse, van Egmond, Noordam, Mulder, & De Nijs, 2014; Marín-Sáez, Romero-González, & Garrido Frenich, 2019). On the other hand, the production of infant cereals consists in different process operations such as ingredient mixing, slurry pre-cooking, drying, milling and packaging. Thermal treatment during slurry pre-cooking is aimed to pasteurize or hygienize the slurry, as well as at achieving complete gelatinization of the starch granules, which is essential for the processing of a ready to reconstitute infant cereals (Gantwerker, Company, & Jones, 1984). This operation consists in treating a mix of cereal flours and water (between 60 and 80 %, w/w) at less than 100 °C for several minutes (Gantwerker et al., 1984; Pascari, Marín, Ramos, Molino, & Sanchis, 2019). Subsequently, the gelatinized slurry is dried above 100 °C, usually in a drum-dryer, which induces an immediate evaporation of the water resulting in a thin dry film of the product (Fernández-Artigas, Guerra-Hernández, & García-Villanova, 1999).

Certain TAs show considerable stability to thermal treatments (Casado, Casado-Hidalgo, González-Gómez, Morante-Zarcero, & Sierra, 2023), although little is known considering the wide range of existing TAs. To the best of our knowledge, the effect of pH on the degradation of TAs has not been studied so far.

The aim of this study was to explore whether the modulation of matrix pH during thermal processing of cereal-based infant foods could be a suitable strategy to reduce tropane alkaloids to provide safer products. To this end, this study assessed a wide range of 24 TAs, as potential contaminants, from Datura and Convolvulaceae weeds. The thermal stability (at 80 to 100 °C) was investigated in a buffer system (at pH 4 and 7), as well as at 180 °C in both spiked and naturally contaminated millet flours as model cereal-based matrix.

2. Material and methods

2.1. Reagents and chemicals

Low molecular weight TAs (6-OH-tropinone (TCI Europe, Zwijndrecht, Belgium), nortropinone (TCI), scopine (TRC, Toronto Research Chemicals, Toronto, Canada), scopoline (Carbosynth, Compton, UK), tropine (TCI), tropinone (TCI) and pseudotropine (TRC)), Convolvulaceae-type (convolamine, convolidine, convolvine and fillalbin (Latoxan, Valence, France; Synchem UG, Felsberg-Altenberg, Germany)) and Datura-type (acetylscopolamine (TRC), anisodamine (Phytolab, Vestenbergsgreuth, Germany), anisodine (Phytolab), α-OH-methylatropine (TRC), apoatropine (Synchem UG, Felsberg-Altenberg, Germany), aposcopolamine (TRC), atropine (TCI), homatropine (TCI), littorine (TRC), noratropine (TRC), norscopolamine (TRC), phenylacetoxytropane (TRC) and scopolamine (TCI)), and the internal standards atropine-d_3 and scopolamine-d_3 (CDN Isotopes, Pointe Claire, Canada), were sourced as described previously by Mulder et al., 2016. Purity of the standards, according to the suppliers, ranged from 87 to 100 %.

For the preparation of mobile phases and all sample preparation, ultrapure water was obtained from the Milli-Q® system from Millipore (Bedford, MA, USA). Acetonitrile and methanol were of HPLC gradient-grade from Merck (Darmstadt, Germany). Citric acid, sodium chloride and sodium dihydrogen phosphate were obtained from Merck (Darmstadt, Germany).

2.2. Sample preparation and treatments

2.2.1. Buffer solutions

For the buffer solutions, standard solutions of 250 μg/kg TAs with deuterated atropine and scopolamine as internal standards were prepared in 0.01 M citrate buffer (pH 4) and 0.01 M phosphate buffer (pH 7) and treated at 80 °C or 100 °C in a water bath (Memmert GmbH + Co. KG, Germany) for 10, 30 or 60 min, following a fully factorial experimental design with three replicates for each condition. The selected conditions simulated typical thermal conditions such as boiling, and included the proposed by the OECD guidelines for the testing of chemicals (OECD, 2007).

After treatment, samples at pH 4.00 and 7.00 were diluted to 50 μg/kg with 50- and 6.65-mM ammonia (respectively) to reach pH 10.50 and analysed following the LC-MS/MS protocol described below. TAs standard solutions in 6.65 mM ammonia were used as controls (no treatment).

2.2.2. Solid samples

A millet flour was purchased in a local supermarket. Once ensured that there was no natural contamination of TAs by applying the analytical protocol described below, 4 g of sample were spiked with a mix of TAs standard solutions at 50 μg/kg with a positive displacement pipet. Spiked samples were treated at 180 °C in an air oven (VWR International, United States) for 10, 30 and 60 min with three replicates for each treatment time.

A positive (naturally contaminated) sample of millet flour containing TAs was also treated, in the same experimental conditions.

2.3. TAs analysis

TA analysis was carried out according to Mulder et al. (2016). Briefly, treated samples (4 g) were transferred to polypropylene tubes of 50 mL and 40 μL of internal standard solution (atropine-d_3 and scopolamine-d_3 of 1000 ng/mL in methanol) was added. Forty mL of extraction solvent (methanol/water/formic acid, 75/25/0.4, v/v/v) were added and after 30 min on a rotary tumbler, tubes were centrifuged

for 15 min at 3500 rpm (Beckman Coulter, Brea, CA, USA). Ten mL of clear extract (supernatant)were taken for further clean-up by solid phase extraction (SPE) purification using a Strata X-C (200 mg/6 mL) (Phenomenex, Torrance, CA, USA). The SPE cartridges were conditioned with 6 mL of methanol and equilibrated with 6 mL of 1 % formic acid solution. The cartridges were loaded with 10 mL of extract, washed with 6 mL of 1 % formic acid solution and dried under vacuum. TAs were eluted from the cartridges with 6 mL of 1 % ammonia in methanol. The eluates were evaporated under a nitrogen stream at 50 °C, reconstituted in 500 μL water/methanol (90/10, v/v) by vortexing for 15 s. The reconstituted extracts were filtered using 0.45 μm PTFE 500 μL filter-vials (UniPrep, Whatman, Maidstone, UK).

The chromatographic system consisted of an Acquity UPLC® (Waters, Milford, MA, USA), equipped with a diode array detector (Acquity PDA detector, Waters, Milford, MA, USA), an electrospray (ESI) as a source of ionization and a triple quadrupole mass spectrometer (Acquity TQD, Waters, Milford, MA, USA). The system was controlled by MassLynx 4.1 software (Waters, Milford, MA, USA). Chromatographic separation was achieved in a 150 × 2.1 mm, 1.7 μm particle size BEH C18 column (Waters, Milford, MA, USA). The column temperature was 50 °C, the flow rate 0.4 mL/min and the sample injection volume 5 μL. The mobile phase followed a linear gradient between A (6.65 mM ammonia in water) and B (1.30 mM ammonia in acetonitrile) as follows: 0.0 min 100 % A, 2.0 min 100 % A, 12.0 min 30 % A 70 % B, 12.2 min 100 % A and 15.0 min 100 % A. The ESI was operated in the positive mode, the source temperature was fixed at 135 °C, the capillary voltage was set at 3.0 kV and the desolvation temperature was set at 350 °C. The cone gas (nitrogen) flow rate was 400 L/h and cone voltage was set at 30 V. TAs were determined by MS/MS with multiple reaction monitoring (MRM) using specific transitions for the different TAs.

External calibration for buffer solutions and matrix-matched calibration curves were performed for each TA compound at 0, 10, 50 and 100 μg/kg. The limits of detection (LOD) and quantification (LOQ) were estimated as the signal-to-noise ratio of 3 and 10 respectively (Supplementary Table 1).

2.4. Statistical analysis

Experiments were performed in triplicate. The results were analysed by ANOVA to identify which factors were statistically significant to explain the differences in TA concentrations and by *t*-test to compare if the reduction of one treatment (compared to the initial concentration) was statistically significant with JMP® version 16 (SAS institute, Cary, NC, USA). A p-value < 0.05 was considered significant.

3. Results and discussion

3.1. Effect of pH on tropane alkaloids stability during thermal treatment at 80 and 100 °C in a buffer system

The effect of pH on TA during heat treatment at 80 and 100 °C is summarised in Fig. 1 (more details are provided in Supplementary Figs 1 and 2). In general, TAs were more sensitive to thermal degradation at pH 7 than at pH 4, though only in some cases the effect was statistically significant ($p < 0.05$) e.g., aposcopolamine, convolvine, homatropine, noratropine, scopine, tropine, tropinone and 6-OH-tropinone (results of ANOVA test are shown in Supplementary Table 2). Higher reduction was achieved at 100 °C compared to 80 °C, especially at pH 7, as shown by the significant interaction between pH and temperature for some of the LMW and Datura-type TAs (Supplementary Table 2).

In buffer solution at pH 4 the treatment at 100 °C for 60 min caused an average reduction of 10 % of LMW TAs, ranging from a reduction of 72 % (scopine) to no reduction effect with possible slight formation (tropine) (Fig. 1 and Supplementary Fig. 1). Negative reduction values may indicate a possible formation of certain TAs during thermal treatment from breakdown of Datura-type TAs as previously described by Marín-Sáez et al., (2019, 2019). The high stability of tropine was probably due to the release of the tropane ring by degradation of atropine or other TAs (Marín-Sáez, Romero-González, & Garrido Frenich, 2019).

In the case of convolvulaceae-type TAs, an average reduction of 5 % was observed. Convolamine concentration was reduced by 21 %, while

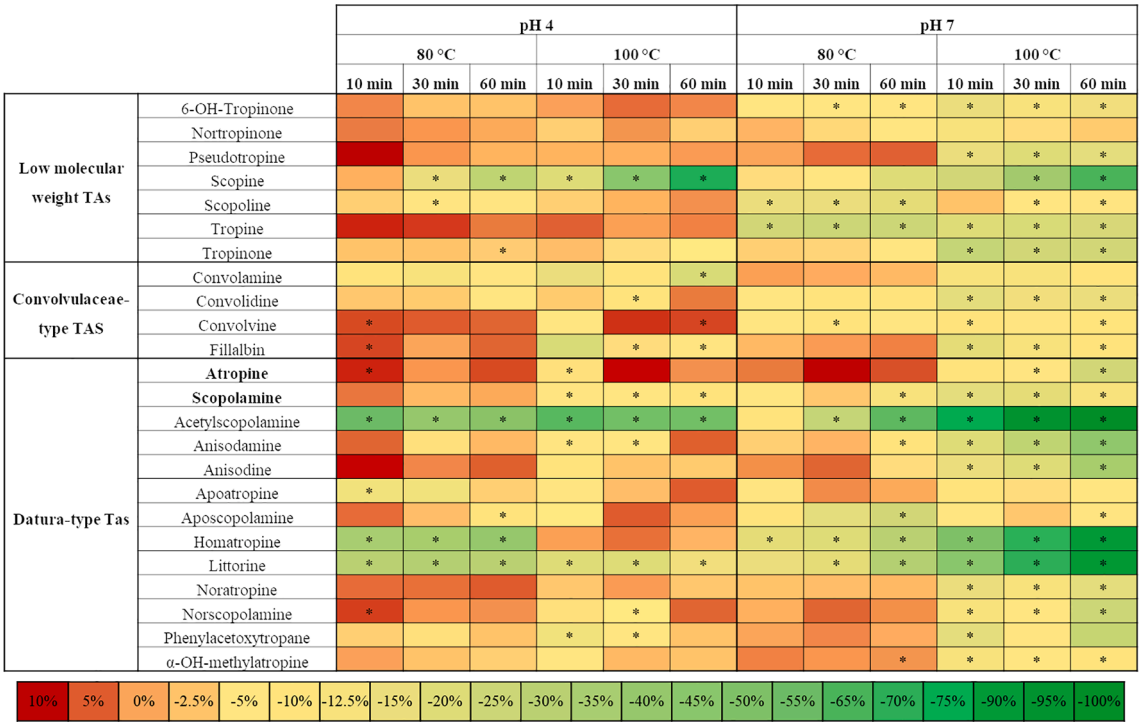

Fig. 1. Heat map of the stability of tropane alkaloids at pH 4 and pH 7 during thermal treatment at 80 and 100 °C. The colour scale describes the different levels of degradation, from lower (red) to higher (green). Asterisks show significant decrease ($p < 0.05$) from the initial concentration.

the relative change of convolvine was −7 %, which suggest the formation of convolvine probably due to convolamine demethylation during thermal treatment (Turgunov, Kadirova, Okmanov, Aripova, & Tashkhodjaev, 2019).

Datura-type TAs were reduced by a 6 % on average. The most heat sensitive Datura-type TA was acetylscopolamines, as its concentration was reduced to the half. In the case of apoatropine, although not increasing significantly from statistical point of view, the high thermal stability could be probably related to its formation by atropine dehydration as descried by Lund and Waaler (1968). Regarding EU regulated TA, maximum degradation of atropine (5 %) and scopolamine (11 %) was achieved at 100 °C (Fig. 1 and Supplementary Fig. 1).

At pH 7, the treatment at 100 °C for 60 min, resulted in an average reduction of the LMW TAs contents of 25 %, ranging from 3 % (nortropine) to 64 % (scopine). Convolvulaceae-type TAs contents decreased by 11 % on average, ranging from 9 % (convolvine) to 15 % (convolidine); while an average of 38 % reduction of Datura-type TAs content, with a range from 5 % (apoatropine) to 100 % (acetylscopolamine), was observed. Contrarily to the observed at pH 4, the higher thermal degradation of TAs at pH 7 overtook all formation reactions, such as those of tropine, convolvine or apoatropine.

The EU regulated TA, atropine and scopolamine, showed remarkable thermal stability. Maximum degradation of atropine and scopolamine was achieved at pH 7 and 100 °C with treatments of 60 and 30 min, respectively. Under these conditions, atropine and scopolamine contents decreased by 23 % and 18 %, respectively (Fig. 1 and Supplementary Fig. 2).

Thermal degradation of TAs at 100 °C has been studied previously in different matrices (Marín-Sáez et al., 2019; Vera-Baquero, Morante-Zarcero, & Sierra, 2022), although this is the first work reporting the effect of pH on a wide range of TAs. Lower or higher degradation was found depending on the matrix and treatment time, with degradation rates between 9 % (aposcopolamine) to 73 % (scopoline) in tea at 100 °C for 6 min; while during the boiling process (100 °C for 10 min) of pasta TAs reduction was between 24 % (apoatropine) and 66 % (tropine). Besides thermal degradation, the reduction was partially attributed to the migration of the TAs to the boiling water. (Marín-Sáez et al., 2019).

In the present study, TAs reached the highest degradation at pH 7. Therefore, during the manufacture of infant cereals, the neutral pH usually occurring during the slurry precooking process close to 100 °C may help reducing TAs contents. However, the high stability shown by some compounds makes the control of TAs by pH not possible if the raw materials are highly contaminated.

3.2. *Effect of 180 °C on tropane alkaloids in millet flour*

Stability of TAs in spiked and incurred commercial millet flour was studied at 180 °C to understand their degradation at a typical high temperature treatment, to assess whether baking or drying at high temperature could be an effective step to reduce TAs content in infant cereal-based food.

Thermal degradation of TAs in spiked millet flour is shown in Fig. 2, and in more detail in Supplementary Fig. 3. LMW TA reduction at 180 °C for 10 min was between 17 % (pseudotopine) and 100 % (scopoline), with an average of 67 %. Longer treatment (60 min); enhanced the TAs reduction to 32 % (tropinone) and 100 % (scopoline), with an average of 75 %. Similar results were observed for Convolvulaceae-type TAs, the reduction at 180 °C for 10 min was between 8 % (fillalbin) and 35 % (convolvine), with an average of 21 %. After 60 min, the reduction was between 44 % (fillalbin) and 94 % (convolvine), with an average

		Spiked millet flour			Naturally contaminated millet flour		
		180 °C			180 °C		
		10 min	30 min	60 min	10 min	30 min	60 min
Low molecular weight TAs	6-OH-Tropinone	*	*	*	NA	NA	NA
	Nortropinone	*	*	*	NA	NA	NA
	Pseudotropine			*	NA	NA	NA
	Scopine	*	*	*	*	*	*
	Scopoline	*	*	*			*
	Tropine	*	*	*	NA	NA	NA
	Tropinone				NA	NA	NA
Convolvulaceae-type TAS	Convolamine		*	*	NA	NA	NA
	Convolidine	*	*	*	NA	NA	NA
	Convolvine	*	*	*	NA	NA	NA
	Fillalbin		*	*	NA	NA	NA
Datura-type Tas	**Atropine**	*	*	*			*
	Scopolamine	*	*	*		*	*
	Acetylscopolamine	*	*	*	NA	NA	NA
	Anisodamine	*	*	*	*	*	*
	Anisodine		*	*	NA	NA	NA
	Apoatropine	*	*	*	NA	NA	NA
	Aposcopolamine	*	*	*	NA	NA	NA
	Homatropine	*	*	*	NA	NA	NA
	Littorine		*	*	NA	NA	NA
	Noratropine	*	*	*	NA	NA	NA
	Norscopolamine		*	*	*	*	*
	Phenylacetoxytropane		*	*	NA	NA	NA
	α-OH-methylatropine			*	NA	NA	NA

10% 5% 0% -2.5% -5% -10% -12 % -15% -20% -25% -30% -35% -40% -45% -50% -55% -65% -70% -75% -90% -95% -100%

Fig. 2. Stability of low molecular weight, Convolvulaceae-type and Datura-type tropane alkaloids in spiked and naturally contaminated millet flour during thermal treatment at 180 °C. The colour scale describes the different levels of degradations observed, from lower (red) to higher (green). Asterisks show significant decrease ($p < 0.05$) from the initial concentration. NA, not applicable (below limit of detection in naturally contaminated millet flour before treatment).

reduction of 71 %. Regarding Datura-type TAs, at 180 °C for 10 min resulted in a reduction between 9 % (littorine) and 100 % (acetylcopolamine), with an average of 32 %; while the reduction after 60 min was between 54 % (phenylacetoxytropane) and 100 % (acetylcopolamine), with an average reduction of 76 %.

Four Datura-type (atropine, scopolamine, anisodamine and norscopolamine) and two LMW TAs (scopoline and scopine) were found in the millet flour from the retail market. Atropine and scopolamine showed the highest values (8.4 ± 1.0 μg/kg and 13.9 ± 1.3 μg/kg, respectively), which were above the EU regulated maximum limits for infant cereals (1 μg/kg). Stability of TAs naturally found in millet flour at 180 °C is shown in Fig. 2 and in more detail in Supplementary Fig. 4.

Among the five TAs identified in commercial incurred millet flour, atropine, scopolamine and scopoline had higher thermal stability in incurred than in spiked flour. In contrast to what was observed in spiked flour, no significant degradation was found for scopolamine after 10 min at 180 °C, nor for atropine and scopoline after 10 and 30 min. The largest difference between spiked and incurred reduction behaviour was shown by scopoline. Scopoline content decreased below LOD after 10 min in spiked flour, while in incurred millet flour showed a maximum reduction of 60 %. On the contrary, norscopolamine content significantly decreased in incurred sample after 10 min, while this compound had no significant decrease in spiked flour. However, after 60 min of treatment, all TAs contents in incurred flour decreased by 63–100 %, similarly to the reduction observed in spiked flour.

Overall results showed a limited effect of thermal treatment at 180 °C on the degradation of TAs in millet flour, especially for short treatment times. Furthermore, the higher thermal resistance found in incurred samples compared with the artificially spiked ones suggests a matrix protection of the millet flour on the degradation of TAs.

There are few studies evaluating the thermostability of TAs in food matrices at temperatures above 100 °C (Marín-Sáez et al., 2019; Vera-Baquero et al., 2022). Vera-Baquero et al. (2022) studied thermal degradation during baking (180 °C, 20 min) in breadsticks prepared with corn flour spiked with seeds of *Datura stramonium* (atropine, scopolamine and anisodamine). The study showed that after 20 min of baking at 180 °C, a degradation of 32 % and 45 % was achieved for atropine and scopolamine, respectively. In the case of anisodamine, degradation was up to 35–49 %, which agrees with the reduction percentages found in the present study for atropine and scopolamine spiked millet flour (33.9 % and 41.8 %, respectively) after 30 min at 180 °C. On the other hand, Marín-Sáez et al. (2019) studied the degradation of TAs in bread made with buckwheat flour contaminated with Solanaceae seeds. The study showed that baking at 190 °C for 40 min induced an 84 % degradation of scopolamine and 73 % of atropine. In general, under the baking conditions described by these authors (190 °C), TAs contents decreased by 94–100 %, which is higher than the observed in the present study with millet flour treated for 30 min at 180 °C.

High occurrence of TAs contamination has been described in cereal-based products, which is in line with TAs concentrations found in the present study in millet flour. Given that TAs concentrations above the maximum levels set by the EU regulation can be found in cereal-based infant food at retail (González-Gómez et al., 2022; Mulder et al., 2016). As heating, including drying process, could not be proved as suitable control measure to ensure TAs concentration below the regulated limit in the EU, TAs represent a chemical hazard that needs to be monitored.

4. Conclusions

The assessment of the effect of pH and temperature as possible process parameters to control TAs content in infant cereal-based food during thermal processing, showed that TAs stability was lower at higher temperature and neutral pH. However, the results suggest that pH and temperature modulation at the range usually applied in the cereal-based infant food manufacture cannot prevent TAs contamination of the final product due to the low rate of thermal degradation. Additionally, food matrix seems to protect TAs from thermal degradation, as observed in naturally contaminated millet flour. Given the existing prevalence of TAs in the raw materials usually used for the manufacture of cereal-based infant food, the findings of the present study highlight the need for an accurate and intensive monitorization of TAs in raw materials in order to prevent contaminated final products.

CRediT authorship contribution statement

Berta Torrents-Masoliver: Conceptualization, Data curation, Writing – original draft, Writing – review & editing. **Carlos Terriente-Palacios:** Formal analysis, Methodology, Validation. **Sara Bover-Cid:** Conceptualization, Funding acquisition, Supervision, Writing – review & editing. **Anna Jofré:** Conceptualization, Supervision, Writing – review & editing. **Massimo Castellari:** Conceptualization, Supervision, Writing – review & editing. **Albert Ribas-Agustí:** Supervision, Writing – review & editing, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

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