PRODUCTION AND CHARACTERISATION OF BIODEGRADABLE FOAM FROM GADUNG TUBER (*Dioscorea hispida* Dennst) STARCH: DETOXIFICATION EFFICACY AND MATERIAL PROPERTIES FOR SUSTAINABLE PACKAGING

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Abstract

Gadung tuber (*Dioscorea hispida* Dennst) starch was evaluated as a raw material for the production of biodegradable foam with safe residual cyanide levels. The initial hydrogen cyanide (HCN) concentration in the extracted starch was 4.01 mg kg^{-1} (dry solid basis). A pH-stability study demonstrated optimum retention of HCN (92.5%) at pH 4 in aqueous slurry, while near-neutral and alkaline conditions led to significant HCN loss through enzymatic and non-enzymatic breakdown of cyanogenic glycosides. Thermal processing by hot-mould baking at 180 °C for 7 min reduced the HCN concentration to 0.54 mg kg^{-1} in the finished foam (dry solid basis), corresponding to an overall detoxification rate of 86.5% relative to the pH-4 slurry (3.71 mg L^{-1}). The optimal foam formulation (starch:water:glycerol:guar gum:magnesium stearate = 75:138:25:1:1, w/w) produced an elastic, homogeneous foam. Soil-burial testing over 9 days showed progressive biodegradation, with mass losses of 7.39%, 15.69% and 21.49% at days 3, 6 and 9, respectively. FT-IR analysis revealed O–H (3330 cm^{-1}) and C–O (1151 cm^{-1}) absorption bands characteristic of starch-based materials, with no detectable nitrile (C≡N) signal. The final HCN concentration of 0.54 mg kg^{-1} is well below the Codex Alimentarius maximum level of 10 mg kg^{-1} for edible cassava-type flours, indicating that the residual cyanide burden is well below the most relevant available benchmark and supporting the potential of this foam as a sustainable packaging material pending formal food-contact migration assessment. The process demonstrates that conventional thermal foam baking not only produces a functional biodegradable material but also effectively detoxifies cyanogenic starch.

Keywords: *Dioscorea hispida*, biodegradable foam, hydrogen cyanide detoxification, starch-based packaging, thermal processing

I. INTRODUCTION

The widespread use of conventional petroleum-derived plastic packaging materials has led to serious environmental pollution because of their long-term persistence in soil and aquatic ecosystems (Tapia-Blácido et al., 2022; Cruz-Tirado, et al., 2019). In response, starch-based biodegradable foams (biofoams) have gained increasing attention as renewable and compostable alternatives for short-life-cycle packaging applications, including disposable food trays and protective packaging (Tapia-Blácido et al., 2022; Cruz-Tirado, et al., 2019). Starch is an attractive raw material because it is abundant, renewable, low-cost and possesses thermoplastic properties when plasticised under thermomechanical processing (Tapia-Blácido et al., 2022; Tarique, et al., 2021). Various fabrication methods, including hot-mould baking, extrusion, thermopressing and microwave heating, have been explored to produce starch-based foams (Tapia-Blácido et al., 2022; Cruz-Tirado, et al., 2019; Engel, et al., 2019; Kaisangsri, et al., 2014).

Gadung tubers (*Dioscorea hispida* Dennst) represent an underutilised starch source in Indonesia. They contain approximately 35–40% starch on a fresh-weight basis (Hazrati, et al., 2021; Mutmainna, et al., 2024) and are widely available throughout Southeast Asia, yet their use is limited by the presence of cyanogenic glycosides (chiefly linamarin) that release hydrogen cyanide (HCN) upon tissue disruption (Mutmainna, et al., 2024; Estiasih, et al. 2022). Traditionally, communities employ indigenous methods: soaking, boiling, fermentation, sun-drying and treatment with wood ash to detoxify gadung tubers before consumption (Mutmainna, et al., 2024; Estiasih, et al. 2022; Estiasih, et al. 2020).

The presence of cyanogenic compounds is a concern for both food safety and material processing, but also represents an opportunity:

if processing conditions can be optimised to achieve both material functionality and effective detoxification, gadung starch could become a valuable feedstock for biodegradable packaging. Importantly, even high-cyanogen cassava varieties can be made safe through careful processing (Nambisan, 2011: Bradbury, 2006). Detoxification proceeds by enzymatic hydrolysis of cyanogenic glycosides via β-glucosidase (linamarase) to acetone cyanohydrin, followed by spontaneous breakdown above pH 5 to free HCN, which is then removed by volatilisation during heating or by dissolution in processing water (Bradbury, 2006; Bradbury, et al., 2011).

Despite extensive work on starch-based foams from cassava, corn and sago (Tapia-Blácido et al., 2022; Cruz-Tirado, et al., 2019; Engel, et al., 2019; Kaisangsri, et al., 2014; Hazrati, et al., 2021), no systematic study has examined whether conventional hot-mould foam baking simultaneously achieves adequate detoxification of cyanogenic starch and produces a foam with acceptable physical properties. Therefore, the present study was designed to investigate: (i) the HCN concentration in extracted gadung starch; (ii) the effect of pH on HCN stability in starch slurry; (iii) the efficiency of HCN removal during thermal foam processing; and (iv) the physical properties, biodegradability and functional-group characteristics of the resulting foam. The overarching goal is to reduces the cyanide burden to a level consistent with internationally recognised safety benchmarks, supporting the candidacy of gadung starch as a feedstock for biodegradable packaging foam.

II. METHODS

Materials and Equipment

Fresh gadung tubers (*Dioscorea hispida* Dennst) were collected from Bu'ne Village, Minasatene District, Pangkep Regency, South Sulawesi, Indonesia. Key reagents included KCN (p.a., Loba Chemie), glycerol (technical grade), magnesium stearate (Faci), guar gum (Lucid) and ninhydrin (Merck-Millipore); 1 M HCl and 1 M NaOH were used for pH adjustment. HCN was quantified on a Cary 50 Conc UV-Vis spectrophotometer (Varian) and functional groups were characterised by a Thermo Fisher Scientific Nicolet™ FT-IR spectrometer (ATR mode). Standard laboratory glassware, a blender, Büchner filtration apparatus and an analytical balance (±0.0001 g) were also employed.

Starch Extraction

Gadung tubers (5 kg) were peeled, washed thoroughly under running water and cut into small pieces. The pieces were blended with distilled water (1:2 w/v) and filtered through cheesecloth. The filtrate was adjusted to pH 4 with 1 M HCl and allowed to stand for 5 h to sediment the starch. The starch precipitate was collected by Büchner filtration and dried in an oven at 80 °C for 8 h. The acid adjustment to pH 4 was selected because preliminary experiments (Section 3.2) showed maximum retention of HCN in the slurry at this pH, ensuring that subsequent thermal removal could be quantified accurately.

HCN Determination by the Ninhydrin Method

Quantitative determination of HCN was performed using the ninhydrin-based spectrophotometric assay described by Surleva et al., (2013), which has been demonstrated to be fast, inexpensive and environmentally benign, and is effective for quantifying cyanide released from plant materials. A calibration series was prepared from a 1000 mg L^{-1} KCN stock solution by serial dilution to 0.04, 0.08, 0.1, 1.0, 3.0 and 5.0 mg L^{-1} in 50 mL volumetric flasks; a blank (0.00 mg L^{-1}) was included. From each standard, 6 mL was transferred to a test tube, mixed with 1 mL of freshly prepared ninhydrin reagent (2% w/v in 2% Na_2CO_3) and 1 mL of 5% Na_2CO_3) solution, and allowed to react for 15 min at ambient temperature. The wavelength of maximum absorption (λ_{max}) was determined by scanning between 400 and 600 nm; all subsequent measurements were performed at 482 nm, in agreement with Surleva et al., (2013). HCN concentrations of unknown samples were interpolated from the linear regression of the calibration curve. All analyses were performed in triplicate.

HCN Stability as a Function of pH

Aliquots of starch (5 g) were dissolved in 250 mL of distilled water, filtered, and the pH of

the filtrate was adjusted to 2, 4, 6, 8, 10 or 12 with 1 M HCl or 1 M NaOH. From each pH-adjusted solution, 6 mL aliquots were analysed for HCN as described in Section 2.3. Triplicate measurements were performed for each pH value, and results are expressed as the mean ± standard deviation. The percentage retention of HCN at each pH was calculated relative to the concentration measured in the dried starch (4.01 mg kg^{-1}, dry solid basis).

Foam Synthesis

Foam formulations were optimised in preliminary trials by varying starch mass (10, 20, 30 g) and glycerol volume (5, 10, 15 mL) while keeping water (50 mL), guar gum (0.4 g) and magnesium stearate (0.4 g) constant. The composition that gave a coherent, elastic foam was selected (corresponding to a mass ratio of starch : water : glycerol : guar gum : magnesium stearate = 75 : 138 : 25 : 1 : 1, w/w). The ingredients were mixed, the slurry was adjusted to pH 4 with 1 M HCl, stirred for 10 min, poured into the aluminium mould, and baked in a preheated oven at 180 °C for 7 min. After cooling at ambient temperature for 30 min, the foam was demoulded. For HCN determination, a 5 g sample of the foam was extracted with 250 mL of distilled water for 30 min with stirring, filtered, and the filtrate was analysed using the ninhydrin method (Section 2.3).

Biodegradability Testing

Foam samples (≈2 × 2 × 0.5 cm) of known initial weight (W_1) were buried in polyethylene containers filled with garden soil (moisture content ≈ 25% w/w) maintained at ambient temperature (28 ± 2 °C). Samples were retrieved after 3, 6 and 9 days, gently cleaned to remove adhered soil, dried at 50 °C to constant mass, and reweighed (W_2). Mass loss was calculated as:

$$\textit{Mass loss (\%)} = [(W_1 - W_2)/W_1] \times 100$$

Triplicate samples were used at each time point.

FT-IR Analysis

Functional groups present in the foam were identified by FT-IR. Spectra were recorded over the range 4000–400 cm^{-1} at a resolution of 4 cm^{-1}, with 32 co-added scans per spectrum.

III. RESULT AND DISCUSSION

Result

HCN Concentration in Native Gadung Starch

The HCN concentration in the dried gadung starch was 4.01 mg kg^{-1} (dry solid basis). This value is considerably lower than the 50–400 mg kg^{-1} reported for fresh gadung tubers (Estiasih, et al. 2022; Estiasih, et al. 2020), indicating that substantial HCN was lost during extraction and drying. The reduction is consistent with two well-documented mechanisms: (i) enzymatic hydrolysis of cyanogenic glycosides by endogenous β-glucosidase (linamarase), which is activated upon tissue disruption and converts linamarin to acetone cyanohydrin; and (ii) subsequent spontaneous decomposition of acetone cyanohydrin above pH 5 to free HCN, which volatilises during oven drying at 80 °C (Bradbury, 2006; Bradbury, et al., 2011). Bradbury and colleagues have shown that combinations of crushing, wetting and sun-drying can remove up to 95% of the cyanogens in cassava roots (Bradbury, 2006; Nambisan, 2011; Cumbana, et al., 2007), and analogous physico-chemical pathways operate during gadung-starch extraction (Mutmainna, et al., 2024; Estiasih, et al. 2022). From a process-safety standpoint, this initial reduction is beneficial because it lowers the cyanide load before foam manufacture.

Effect of pH on HCN Stability in Starch Slurry

The stability of HCN in the starch slurry was strongly pH-dependent (Table 1). The highest HCN concentration (3.71 mg L^{-1}) was observed at pH 4, corresponding to a retention of 92.5% relative to the dried starch. At pH 2 the retention was slightly lower (90.0%). At pH 6 and 8 the HCN concentration dropped sharply to ≈1.3 mg L^{-1} (only 31–33% retention). At pH 10 and 12 the cyanide–ninhydrin adduct underwent a colour change (red → blue) attributable to deprotonation of the chromophore under strongly alkaline conditions, rendering absorbance measurements at 482 nm unreliable for

quantitative work; these values are therefore not reported. The blue adduct ($\lambda_{max} \approx 590$ nm) is less stable and has a lower molar absorptivity than the red form, so quantitation at pH > 9 would require recalibration at the alternative wavelength (Surleva et al., 2013).

These findings indicate that HCN is most stable under weakly acidic conditions. Two complementary mechanisms account for this behaviour. First, at pH values well below the pK_a of HCN (9.21 at 25 °C), the undissociated molecular form predominates; this species is less reactive toward nucleophilic attack and less susceptible to oxidative or hydrolytic loss than the cyanide anion (Nambisan, 2011: Bradbury, 2006). Second, acidic conditions suppress the activity of linamarase (optimum pH ≈ 5–6) and slow the spontaneous decomposition of acetone cyanohydrin (which is rapid above pH 5), thereby preserving any intact cyanogenic species in the slurry (Bradbury, 2006; Bradbury, et al., 2011; Cumbana, et al., 2007). The pH-stability data therefore confirm that HCN can be retained in the wet slurry when needed (for analytical quantitation), but they also imply that the subsequent thermal step must be designed to maximise HCN removal—an outcome demonstrated in Section 3.3

Table 1. HCN concentration in starch slurry as a function of pH.

pH	Absorbance (482 nm)	HCN (mg L^{-1})	Retention (%)
2	0.1135 ± 0.0003	3.61 ± 0.01	90.0
4	0.1174 ± 0.0004	3.71 ± 0.01	92.5
6	0.0286 ± 0.0005	1.31 ± 0.02	32.7
8	0.0259 ± 0.0004	1.24 ± 0.02	30.9
10	n.d.[a]	n.d.[a]	n.d.[a]
12	n.d.[a]	n.d.[a]	n.d.[a]

[a] n.d. = not determined; at pH ≥ 10 the cyanide–ninhydrin adduct shifts from red to blue ($\lambda_{max} \approx 590$ nm) and absorbance at 482 nm is no longer proportional to HCN concentration (Surleva et al., 2013)

Detoxification During Thermal Foam Processing

After hot-mould baking at 180 °C for 7 min, the HCN concentration in the final foam was 0.54 mg kg^{-1} (dry solid basis). Relative to the pH-4 slurry (3.71 mg L^{-1} in the aqueous slurry), this corresponds to an 86.5% reduction during the thermal step. For the purpose of this cross-phase comparison, the slurry concentration (3.71 mg L^{-1}) is treated as numerically equivalent to 3.71 mg kg^{-1} on the basis that the density of the dilute aqueous slurry is approximately 1 kg L^{-1}; the resulting 86.5% reduction therefore represents a conservative estimate. The result is consistent with the well-established principle that free HCN is highly volatile (boiling point 25.6 °C) (National Center for Biotechnology Information (NCBI), 2024). and is rapidly lost when cyanogenic materials are heated under conditions that also disrupt the cellular matrix (Nambisan, 2011: Bradbury, 2006; Bradbury, et al., 2011; Cumbana, et al., 2007). Comparable removal efficiencies (>80%) have been reported for cassava processed by sun-drying combined with crushing (Nambisan, 2011: Bradbury, 2006), by the wetting method (Bradbury, 2006; Cumbana, et al., 2007) and by extrusion-cooking (Cuevas-Carballo, 2017).

The final concentration of 0.54 mg kg^{-1} is well below the Codex Alimentarius maximum tolerable level of 10 mg HCN kg^{-1} established for edible cassava flour (CODEX STAN 176-1989) and adopted internationally as the reference standard for cyanogenic plant foods (Codex Alimentarius Commission, 1995). The Joint FAO/WHO Expert Committee on Food Additives (JECFA) has further concluded that exposure at the 10 mg kg^{-1} level is not associated with acute toxicity (Joint FAO/WHO Expert Committee on Food Additives (JECFA), 2012). By comparison, the United States Environmental Protection Agency maximum contaminant level (MCL) for free cyanide in drinking water is 0.2 mg L^{-1} (United States Environmental Protection Agency (US EPA)., 2024), and although direct comparison between food and drinking-water standards is imperfect, both reference values indicate that the residual cyanide in the present foam is at a level of negligible toxicological concern, particularly given that the foam is intended for packaging rather than ingestion. The thermal foam-baking step therefore, acts as both a structuring and a detoxification operation. Although comprehensive food-contact safety would require migration testing

under standardised conditions (e.g., EU Regulation 10/2011), the residual HCN level of 0.54 mg kg^{-1} (dry foam basis) is encouragingly low and consistent with the available toxicological benchmarks, warranting further investigation of gadung-starch foam as a packaging material.

Physical Properties and Biodegradability of the Foam

Among the formulations tested, the composition starch : water : glycerol : guar gum : magnesium stearate = 75 : 138 : 25 : 1 : 1 (w/w) produced an elastic, homogeneous and cohesive foam with no visible cracks. Formulations containing less starch (10–20 g per batch) failed to form a coherent foam and remained gel-like, while formulations with insufficient glycerol (5 mL per batch) produced brittle, friable foams. Glycerol functions as a plasticiser, intercalating between starch chains and disrupting hydrogen-bond networks among amylose and amylopectin, thereby reducing brittleness and improving flexibility, (Tarique, et al., 2021; Mutmainna et al., 2023) guar gum and magnesium stearate act, respectively, as a thickener that stabilises the foam cell structure during expansion and as a lubricant that prevents adhesion to the mould (Engel, et al., 2019; Kaisangsri, et al., 2014; Lawton, et al., 2024).

Soil-burial testing demonstrated progressive degradation over the 9-day observation period, with mass losses of 7.39%, 15.69% and 21.49% after 3, 6 and 9 days, respectively. These values are comparable to those reported for cassava- and corn-starch foams under similar conditions (Cruz-Tirado, et al., 2019; Engel, et al., 2019; Kaisangsri, et al., 2014; Polat et al., 2013), and indicate that the gadung-starch foam undergoes microbial attack and hydrolytic depolymerisation when exposed to a moist soil environment. The progressive degradation rate is consistent with the role of the hydrophilic starch matrix as a microbial substrate and with the gradual disruption of the foam cell walls by soil moisture (Tapia-Blácido et al., 2022; Cruz-Tirado, et al., 2019). Complete biodegradation of similar starch foams has been reported within 7–12 weeks under aerobic composting conditions (Polat et al., 2013), suggesting that the present material is suitable for short-term packaging applications followed by composting after disposal.

FT-IR Spectroscopy

The FT-IR spectrum of the bioplastic foam exhibited four diagnostic absorption bands. A broad band centred at 3330 cm^{-1} is assigned to O–H stretching vibrations of hydroxyl groups in amylose, amylopectin, glycerol and absorbed water (Tarique, et al., 2021; Mutmainna et al., 2023; Kizil, et al., 2002). The band at 2932 cm^{-1} corresponds to C–H stretching of aliphatic $–CH_2–$ groups in the polysaccharide backbone and in the glycerol plasticiser (Tarique, et al., 2021; Mutmainna et al., 2023). The intense band at 1151 cm^{-1} is attributed to C–O stretching of the α-1,4 and α-1,6 glycosidic linkages in starch (Mutmainna et al., 2023; Kizil et al., 2002), and the band at 680 cm^{-1} arises from C–H out-of-plane bending. These assignments are fully consistent with previous reports for starch-based films and foams (Tarique, et al., 2021; Mutmainna et al., 2023; Polat et al., 2013, Kizil, et al., 2002). Crucially, no absorption was detected in the 2200–2050 cm^{-1} region, where the C≡N stretch of nitrile or cyanate species would appear, in agreement with the very low residual cyanide level (0.54 mg kg^{-1}, dry foam basis) determined by the ninhydrin assay.

Discussion

Two principal findings emerge from this study. First, conventional hot-mould baking achieves an overall HCN reduction of approximately 87% within a single 7-min processing step, taking the cyanide level from 3.71 mg L^{-1} in the pH-4 slurry to 0.54 mg kg^{-1} in the finished foam (dry solid basis). Because the residual concentration is more than an order of magnitude below the Codex Alimentarius reference value of 10 mg kg^{-1} for edible cassava flour (Codex Alimentarius Commission, 1995; Joint FAO/WHO Expert Committee on Food Additives (JECFA), 2012), the process effectively eliminates the principal toxicological barrier to the industrial use of gadung starch. The pH-dependence study (Section 3.2) further indicates that careful control of the slurry pH allows the

processor to manage HCN behaviour at each stage: acidic pH stabilises HCN for monitoring or recovery, while the elevated baking temperature drives volatilisation.

Second, the resulting foam exhibits desirable properties for short-life packaging: it is elastic, homogeneous and biodegradable in soil within a few weeks of disposal. The foam can be produced using equipment already common in the starch-foam industry (Engel, et al., 2019; Kaisangsri, et al., 2014; Lawton, et al., 2024. Polat et al., 2013), suggesting a feasible pathway to commercial implementation that requires neither additional detoxification pretreatment nor specialised infrastructure.

Several limitations of the present work should be acknowledged. The mechanical properties (tensile strength, compressive strength, density and water-absorption capacity) were not quantified; these parameters are essential for benchmarking against expanded polystyrene and other commercial foams (Polat et al., 2013) and should be addressed in follow-up studies. The biodegradation test was limited to 9 days and to soil burial; standardised assays under controlled composting conditions (e.g., ISO 14855) and aqueous biodegradation (ISO 14852) would more rigorously establish the end-of-life behaviour of the material (Polat et al., 2013). Hydrolytic resistance and moisture sensitivity, well-known weaknesses of unmodified starch foams (Polat et al., 2013; Soykeabkaew et al., 2015), should also be quantified. Finally, the ninhydrin assay, while validated and inexpensive (Surleva et al., 2013), is sensitive to pH extremes; future studies should confirm HCN results with an orthogonal technique such as ion chromatography with pulsed amperometric detection or gas chromatography–mass spectrometry. It should also be noted that the Codex Alimentarius limit cited here applies to edible cassava flour for ingestion, not to packaging materials; a formal food-contact safety assessment would require migration testing of HCN and other constituents from the foam matrix into food simulants under realistic contact conditions. Morphological characterisation of the foam, in particular, scanning electron microscopy (SEM) of cryo-fractured cross-sections to quantify pore size, pore size distribution, cell wall thickness and the ratio of open to closed cells, was not performed in the present study. This is a significant limitation, since pore structure is a primary determinant of both biodegradation rate (through its effect on accessible surface area and water penetration) and mechanical performance (through its influence on compressive modulus and fracture resistance) [refs]. Future studies should include SEM analysis and should seek to establish quantitative structure–property relationships linking foam microstructure to biodegradation kinetics and mechanical behaviour.

From a process-design standpoint, the present results suggest that gadung starch can be substituted directly into existing starch-foam manufacturing lines without an additional detoxification pretreatment. This finding supports the valorisation of an underutilised, locally available and low-cost agricultural resource for sustainable packaging. Promising avenues for future work include the incorporation of lignocellulosic fillers (e.g., cassava peel, sugarcane bagasse, banana stalk) to improve water resistance and mechanical performance (Polat et al., 2013, Machado, 2017), and pilot-scale evaluation of energy and mass balances to confirm industrial viability.

IV. Conclusion

Gadung tuber (*Dioscorea hispida* Dennst) starch was successfully processed into a biodegradable foam by conventional hot-mould baking. The initial HCN concentration of the extracted starch was 4.01 mg kg^{-1} (dry solid basis), and the baking step (180 °C, 7 min) reduced the HCN content by 86.5% relative to the pH-4 slurry (3.71 mg L^{-1}), yielding a final foam concentration of 0.54 mg kg^{-1} (dry foam basis). This level is more than an order of magnitude below the Codex Alimentarius maximum of 10 mg kg^{-1} for edible cassava-type flours, confirming that thermal foam baking simultaneously produces a functional material and effectively detoxifies the cyanogenic starch. The optimal formulation (starch : water : glycerol : guar gum : magnesium stearate = 75 : 138 : 25 : 1 : 1, w/w) gave an elastic, homogeneous foam with progressive biodegradability under soil burial (7.39–21.49% mass loss over 9 days). FT-IR analysis confirmed the characteristic O–H, C–H and C–O absorptions of starch-based materials and the absence of nitrile signals. Gadung starch therefore represents a

promising and currently underutilised feedstock for biodegradable packaging foam, with a residual HCN level well below relevant toxicological benchmarks; full food-contact safety confirmation will require standardised migration testing in future work.

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