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Synthesis and Characterization of Difurfurylidene Triurea Obtained by Heterogeneous and Solution Methods from Non-Distilled Furfural

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Abstract: The condensation of urea with furfural produces difurfurylidene triurea (DFTU). DFTU can be used in agriculture as a slow-release fertilizer. Its general behavior aligns with that of synthetic organic slow-release fertilizers. The two products, urea and furfural, released in the soil have different beneficial effects. Urea acts as a fertilizer, and furfural acts as a fungicide and nematicide. This work aims to verify that DFTU can be obtained using technical (non-distilled) furfural and to estimate the time required for DFTU hydrolysis. In this study, samples were prepared by two synthesis methods: the heterogeneous method and the solution method. The products were characterized using FTIR, XRD, SEM, and BET techniques. The kinetic study was conducted using two approaches: determining furfural concentration by HPLC and analyzing total nitrogen content by the Kjeldahl method. Results showed that DFTU formation is independent of the initial urea: furfural molar ratio and whether distilled or non-distilled furfural is used. The kinetic study demonstrated that the in vitro hydrolysis rate of DFTU depends on the synthesis method. The product obtained in solution hydrolyzes 1.7 times faster than DFTU prepared in bulk. Based on the hydrolysis rate of DFTUH-D, it is expected that the product releases urea and furfural for more than 9 months in vitro. This study could be a valuable alternative for the agricultural industry, considering the different crop cycles in which it may be used as a slow-release fertilizer.

1. Introduction

The condensation of urea with furfural produces difurfurylidene triurea (DFTU). Its synthesis can be performed both in solution and in bulk without significant changes in the product characteristics (Martínez-García *et al.,* 2004). Like other condensation compounds of urea with aldehydes, such as urea-formaldehyde (ureaform), either as a pure product or enriched with other components (Rivera *et al.,* 2021), and isobutylidene diurea, DFTU can be used in agriculture as a slow-release fertilizer by releasing urea through hydrolysis caused by rain or irrigation (García-Gómez *et al.,* 1999). The general behavior of DFTU aligns with that of synthetic organic slow-release fertilizers (Priya *et al.,* 2024). Still, it stands out because the two products released into the soil, urea and furfural, had distinct beneficial effects. Urea acts as a fertilizer, whereas furfural has been reported to exhibit fungicidal and nematicidal properties. Owing to their slow release, these compounds may be more efficiently utilized by plants. In contrast to conventional fertilizers and pesticides, which are often applied indiscriminately and may contribute to soil and groundwater contamination (Jghalef B., 2016) as well as postharvest losses such as rapid vegetable decay (Sanou Y., 2024), these materiales offer a potentially more controlled alternative.

The fungicidal action of furfural has been known for a long time. As early as 1926, a patent described its effectiveness as an inhibitor of wheat bunt (Tilletia foetida) growth, noting that seed treatment with furfural does not affect germination (Miner, 1926). Good results have been reported using furfural against other important phytopathogenic fungi, such as A. mali (Jung *et al.,* 2007) and Rhizoctonia solani (El-Mougy *et al.,* 2012).

Furfural's nematicidal action is also well-known. It is considered an effective bionematicide with an EC50 value of 24 µg/mL (Kumar *et al.,* 2023). It produces good results against the genus Meloidogyne (root-knot nematodes), one of the most destructive plant nematodes (Theofilidou *et al.,* 2023). Furfural has been effectively used against Meloidogyne spp. in cotton (Bauske *et al.,* 1994), tomato (El-Mougy *et al.,* 2008), sunflower (Mohamed, 2017), and soybean (Rodriguez-Kabana *et al.,* 1993). It is also effective against Meloidogyne arenaria and Pratylenchus brachyurus in zucchini cultivation and against Heterodera glycines and other species when soybean (Glycine max) is used as a host plant. Good results were obtained against Meloidogyne arenaria and Paratrichodorus minor in okra crops (Zeitsch, 2000) and against Meloidogyne incognita (Adenike, 2020). In sugarcane cultivation, replacing the highly toxic commercial nematicide carbofuran with furfural has been recommended (Fabiyi, 2021).

Furfural is the active ingredient in commercial nematicides CropGuard® and Protect® (both from Illovo Sugar South Africa Limited, Merebank, Durban, South Africa) and MultiGuard Protect® (Agriguard Company, LLC, Cranford, New Jersey, USA) (Fourie *et al.,* 2014).

Two main theories explain the nematocidal action of furfural. One is that it affects the nematode cuticle, preventing movement and causing death by dehydration or parasitic organisms (El-Mougy *et al.,* 2008), (Fourie *et al.,* 2014), and it affects fungi by reacting with their cell walls, altering their functions (El-Mougy *et al.,* 2008). The other theory is that furfural does not kill nematodes directly but alters the soil microflora to stimulate rapid growth of antagonistic bacteria that eliminate them. Compared to other nematicides, furfural offers advantages such as similar effectiveness at a lower cost and low toxicity (Zeitsch, 2000).

Furfural condensation with urea could be performed even without industrial purification of furfural. The common practice is to distill furfural before use, which may be dark colored, from yellow to black. However, this should not prevent its use in DFTU synthesis, since the color does not indicate loss of properties. The impurities coloring furfural are typically small. According to Zeitsch (Zeitsch, 2000), a commercial 98% purity product that darkens during storage yields 97% pure furfural when distilled; even more remarkably, furfural stored for years can gel yet still yield 90% pure furfural by distillation. Note that technical furfural is used as a nematicide and fungicide under the commercial product MULTIGUARDTM PROTECT, so impurities do not affect its function.

Our work had two objectives: 1) verify the hypothesis that technical furfural can produce DFTU with similar results to product from distilled furfural for use as a slow-release fertilizer; 2) estimate, through a kinetic study, the time required for DFTU hydrolysis.

2. Methodology

2.1 Synthesis

2.1.1 Synthesis of DFTU in bulk (heterogeneous phase)

DFTU synthesis was performed using purified furfural (DFTUH-D) by reduced-pressure distillation and non-purified furfural (DFTUH-SD). The reaction was carried out in a test tube by adding 2 g of urea and 1.80 mL of furfural. Reactants were mixed until the urea was completely

absorbed by furfural, ensuring maximum interaction. The test tube was placed in a heating bath at 75–80 °C for 4 hours, then removed and left at room temperature for one day. The product was washed three times with ethanol and vacuum filtered. It was then dried in an oven at 50 °C until constant weight.

2.1.2 Synthesis of DFTU in solution

The reaction took place in a beaker where 27 g of urea was dissolved in 30 mL of water under constant stirring. At room temperature, 60 mL of non-distilled furfural was added, and the stirring was increased to 800 rpm. The reaction continued for 2 to 3 hours until a dark-colored paste-like precipitate formed. The reaction was stopped, and the product was dried at room temperature for 24–48 hours, and then transferred to a desiccator for complete drying.

2.2 Experiments

2.2.1 Hydrolysis kinetics of DFTU

Kinetic studies were performed using the following two methods:

1) Furfural concentration by HPLC.

Mass-prepared DFTU from pure reagents was used. In a sealed container, 0.9985 g of DFTU and 100 mL of distilled water were added. Hydrolysis was conducted at 30 °C with gentle stirring (30 min before each sampling). Samples of 1 mL were taken periodically, diluted with 1 mL of distilled water, and analyzed by HPLC with UV detection to quantify released furfural. Calibration curve for signal area (A) vs. furfural concentration (C_F) in mol/L was established: $A=4.08\times10^8 C_F$ ($R=0.99991$; $R^2=0.9998$). Furfural mass (m_F) was calculated from C_F by $m_F=0.25C_F$. The reaction hydrolysis shown in **Figure 1** was considered:

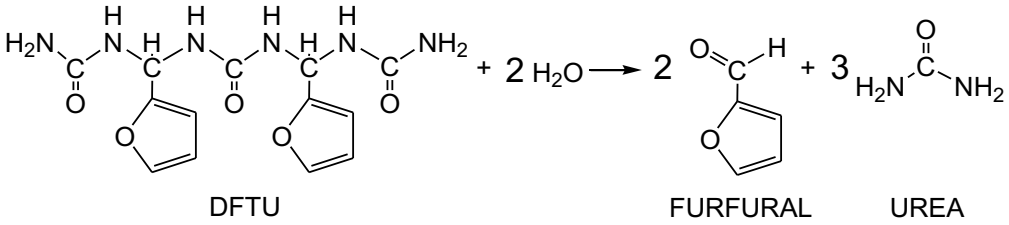

Figure 1. Reaction hydrolysis of DFTU.

Hydrolysis percentage (%H = moles of furfural released) calculated as **Eqn. 1**:

$$\%H = \qquad \textbf{Eqn. 1}$$

$$\%H = 175,1 \times m_F$$

2) Total nitrogen (N_T) by Kjeldahl method.

Hydrolysis kinetics of samples prepared with non-distilled furfural were followed by total nitrogen content (N_T) using the Kjeldahl method and volumetric titration per Cuban fertilizer standard (Norma NC 1121:2016, 2017). For the sample digestion, ~1 g of sample was taken into a 500 mL Kjeldahl flask with 20 mL sulfuric acid (98%) and 10 mL distilled water. The mixture was boiled, cooled, and transferred to a 100 mL flask, volume made up and homogenized. An aliquot of 10 mL diluted sample was transferred to a 750 mL Kjeldahl flask with 300 mL of water and glass beads. In a beaker, 15 mL of 0.1 M sulfuric acid with Tachiro indicator was placed for distillation.

Then, 30 mL of 50% sodium hydroxide solution was added to the flask walls, and the flask was sealed. The mixture was slowly distilled until ~250 mL of distillate was collected. Excess acid was titrated with 0.1 M NaOH until the color changed from violet to light green. Total nitrogen content (as ammoniacal nitrogen) calculated by **Eqn. 2**:

$$N_T(\%) = 1,4 \times \frac{[V(H_2SO_4) \times C(H_2SO_4)] - [V(NaOH) \times C(NaOH)]}{mass} \quad \text{Eqn. 2}$$

Where:
1,4: chemical milliequivalent of N per 100
V (H_2SO_4): volume of sulfuric acid solution (mL)
C (H_2SO_4): concentration of sulfuric acid (mol/L)
V (NaOH): volume of sodium hydroxide solution consumed in titration (mL)
C (NaOH): concentration of sodium hydroxide (mol/L)
mass: sample mass (g)

2.3 Characterisation of DFTU product

The products were characterized using Fourier Transform Infra-Red Spectroscopy (FTIR), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Brunauer-Emmet-Teller (BET) analysis. FTIR (FTIR NICOLET Avatar-670, Madison, USA) was used to determine the functional groups present in the samples. XRD (D-8 Advance, BRUKER, Germany) patterns were recorded at room temperature using Cu-Kα radiation. SEM (JEOL-5600 LV, Japan) was employed to study the surface morphology of the particles. The sample was taken to a sputter coater (EMS 550X) and coated with 20 nm of gold. The acceleration voltage of the microscope was set to 25 kV. A Micromeritics ASAP 2405 N was used to obtain specific surface area by N_2 adsorption/desorption at 77.3 K. Samples were activated for 16 hours. Surface area and average pore size were determined by BET method. Pore size distribution calculated using desorption isotherms and Barret-Joyner-Halenda (BJH) procedure.

3. Results and Discussion

3.1 Synthesis of DFTU

The condensation reaction between urea and furfural was performed by two synthesis routes: heterogeneous phase and solution phase (See **Table 1**).

Table 1. Product yield of DFTU samples for the two synthesis methods employed in this study.

Synthesis Method	Sample	Product Yield
Heterogeneous phase	DFTUH-D	63–66 %
	DFTUH-SD	65-68 %
Solution phase	DFTUAc-SD	68-71%

DFTU synthesis in the heterogeneous phase used both purified and non-purified furfural. Both reactions were conducted simultaneously under identical conditions. Reactants were mixed until urea was fully dispersed in furfural to maximize interaction. The reaction occurs on the urea particle surface, forming a DFTU coating that hinders the unreacted urea's contact with diffusing furfural, slowing the reaction rate.

The solution-phase synthesis used non-purified furfural. For the solution phase method, the reaction was faster than in the heterogeneous phase. The qualitative reaction behaviour using non-distilled furfural matched that of distilled furfural. The marked water insolubility of the product caused a dispersed paste formation in both cases.

3.2 Characterization of DFTU

FTIR spectra of DFTU products are shown in **Figure 2**. The presence of characteristic bands at the same frequencies across all samples indicates: 1) the same product is obtained regardless of synthesis method, and 2) the purity of starting reactants does not alter the product. The first result agrees with previous reports (Martínez-García et al., 2004), and the second is novel.

The IR absorption bands of the solution-prepared sample (DFTUAc-SD) are somewhat broader and less sharp compared to heterogeneous-phase products (DFTUH-D and DFTUH-SD), likely related to differences in product crystallinity.

Key characteristic bands include the amide group with two bands for ν^a NH_2 and ν^s NH_2 at 3450 and 3340 cm^{-1};

- Amide I: C=O stretch, intense band at 1670 cm^{-1} owing to high bond polarity
- Amide II: NH bending (δ_{NH_2} and δ_{NH}), near 1530 cm^{-1}
- Amide III: coupled ν_{CN} and ν_{NH} vibrations, important secondary amide band at 1130 cm^{-1},
- Amide IV: out-of-plane NH bending γ_{NH} at 740 cm^{-1}.

Furan ring presence confirmed by ν_{C-C} bands near 1600 cm^{-1} and γ_{CH} at 740 and 590 cm^{-1}.

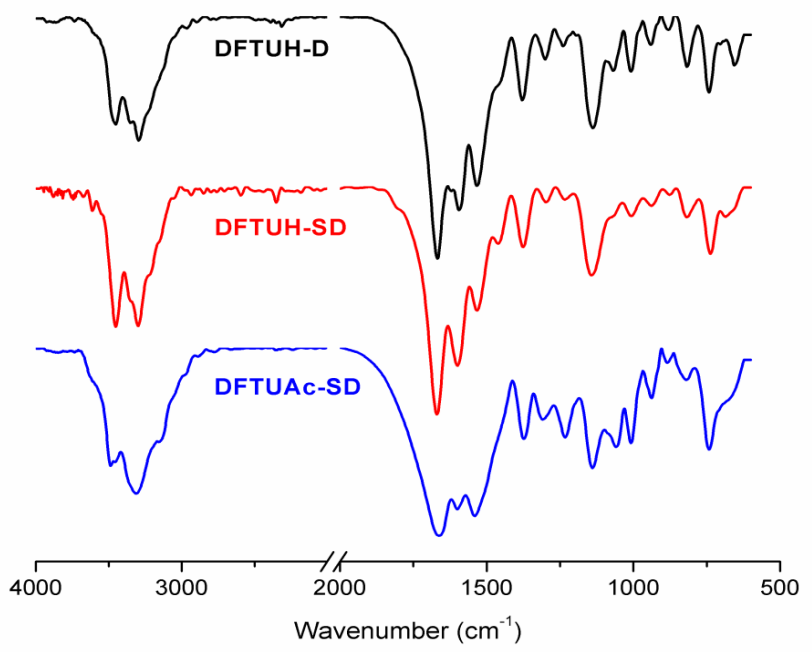

Figure 2. FTIR spectra of DFTU products.

XRD diffractograms of DFTUH-SD and DFTUAc-SD are shown in **Figure 3**. Two intense peaks appear at diffraction planes (2θ = 21° and 23°), consistent with literature (Martínez-García et al., 2004). These highest intensity peaks reveal differences in crystallinity: DFTUH-SD shows higher crystallinity (narrower peak) than DFTUAc-SD, attributed to reaction rate differences. In solution, faster reaction inhibits formation of more crystalline material, while heterogeneous phase reaction's slower rate favors more crystalline structures. SEM results indicate generally micrometric particle

sizes and irregular surfaces for both DFTUH-SD and DFTUAc-SD. Figure 4 shows aggregate formation and irregular granules, some tending to spherical shape especially in DFTUAc-SD.

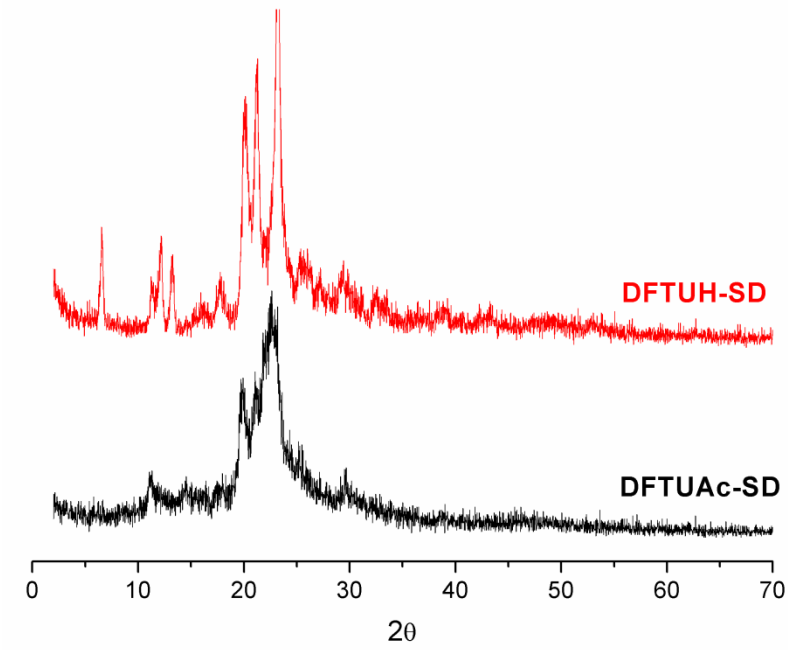

Figure 3. X-ray diffractogram of DFTUH-SD and DFTUAc-SD products.

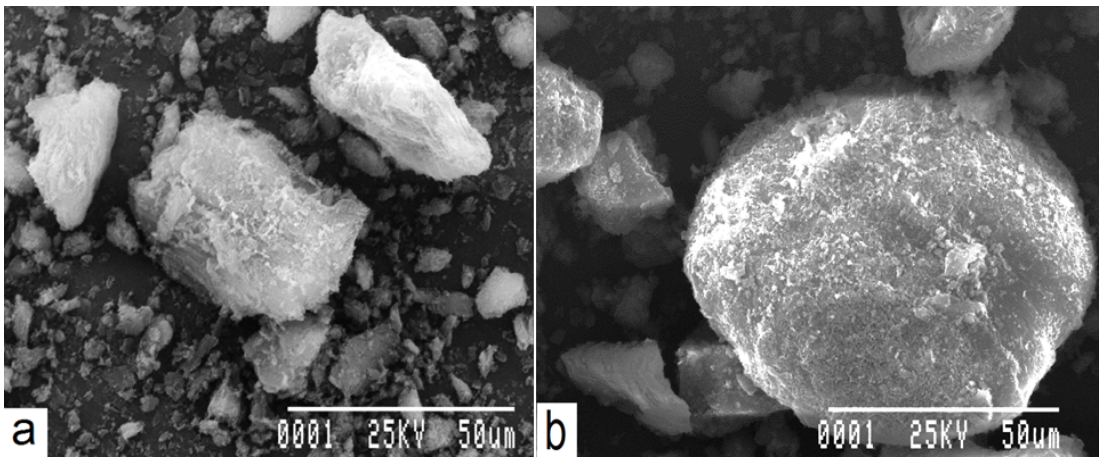

Figure 4. SEM micrograph of (a) DFTUH-SD and (b) DFTUAc-SD.

BET adsorption isotherms show small specific surface areas: 18.01 m^2/g for DFTUH-SD and 7.04 m^2/g for DFTUAc-SD; pore volumes of 0.11 cm^3/g and 0.03 cm^3/g, respectively, indicating very low porosity. Both are mesoporous with pore sizes of 25.2 nm (DFTUH-SD) and 19.4 nm (DFTUAc-SD). These results suggest pore contribution to urea and furfural release is negligible and that hydrolysis occurs at particle surfaces.

3.3 Kinetic study of DFTU hydrolysis

3.3.1 Total nitrogen release from DFTU

An in vitro study via Kjeldahl method determined % total nitrogen (N_T) released from DFTUH-SD and DFTUAc-SD products over 30 days (See **Figure 5**). We can see that the released nitrogen derives entirely from urea, as furfural contains no nitrogen atoms. Both products show a rapid initial

release ("burst effect") of N_T: 4.5% for DFTUH-SD and 7.5% for DFTUAc-SD, linked to nitrogen compounds on the particle surface which are quickly released.

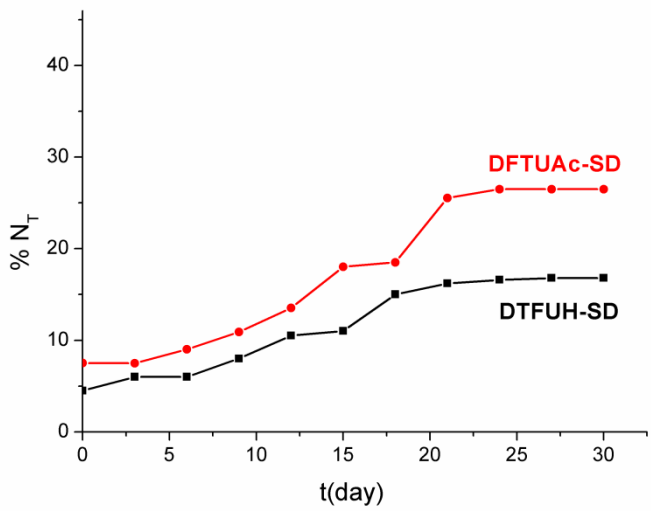

Figure 5. Comparison of % release of N_T for DFTUH-SD and DFTUAc-SD.

Between 2.5 and 25 days, hydrolysis follows zero-order kinetics for both compounds, **Figure 6**.

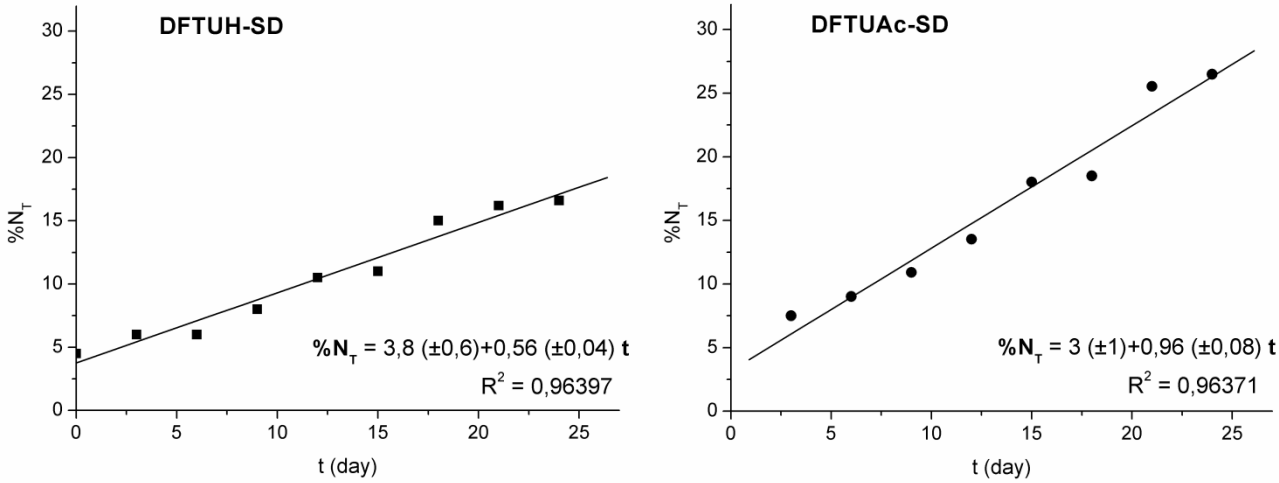

Figure 6. Kinetic study of hydrolysis: DFTUH-SD and DFTUAc-SD.

Although the hydrolysis rate depends on solid-liquid surface area (Grénman et al., 2011), DFTUH-SD did not hydrolyze faster despite its expected higher surface area; both products have similarly small surfaces. The difference is explained by the synthesis method. DFTUAc-SD was prepared in solution with a higher reaction rate impeding crystalline formation, whereas DFTUH-SD formed slowly in the heterogeneous phase, favoring crystallinity.

More crystalline products hydrolyze more slowly than less crystalline ones (Hall et al., 2010). Hence, the zero-order hydrolysis rate is 1.7 times faster for the solution-prepared product than the bulk-prepared product (0.96 vs. 0.56).

3.3.2 Furfural release monitored by HPLC

Slow-release urea fertilizers have low water solubility. Isobutylidene diurea (IBU) solubility is 0.2 g/100 mL at 20 °C (National Center, 2025); ureaform oligomers exhibit solubility from 2.2 g/100

mL to trace amounts depending on molecular size (Hayase et al., 1969). IBU releases urea by soil moisture-induced hydrolysis (ME Trenkel, 2021), and ureaform mainly through microbial action (Lewu et al., 2020). DFTU is even more insoluble than these compounds (no experimental solubility value reported). Its hydrolysis mechanism (chemical vs. biological) is unknown. The kinetic study, though not replicating soil conditions, indicates the potential slowness of urea release.

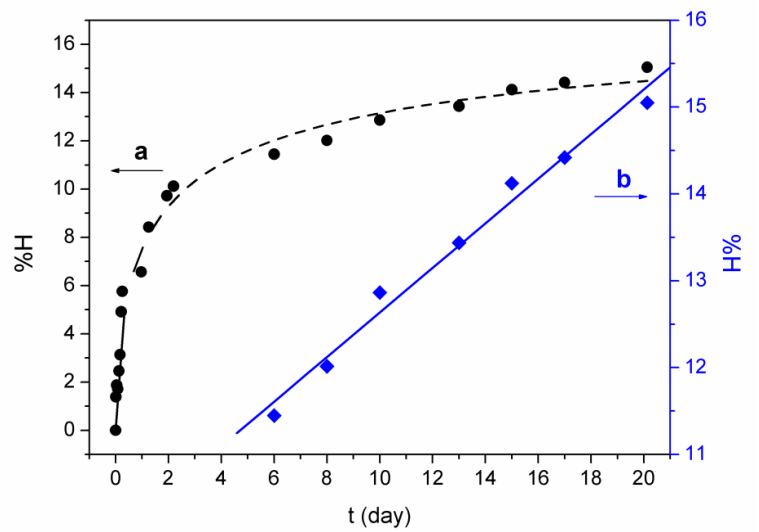

Figure 7. Kinetic study of DFTU hydrolysis: (a) Variation of the percentage of hydrolysis over time and (b) Linear fitting of the curve behaviour from day 6.

The hydrolysis of DFTU obtained in bulk from pure reagents was monitored by observing the change in concentration of furfural in the surrounding water. The increase in the percentage of hydrolysis (%H) is shown in **Figure 7**. Hydrolysis exhibits two phases: an initial rapid stage reaching 10.12% hydrolysis in 2.19 days, and a slower phase progressing 4.92% over 17.93 days. In the slower phase, %H depends linearly on time (Figure 6b). The linear fit equation is: %H = 10.1 (± 0,2)+ 0.26 (± 0,01) t. Extrapolating to 85%, hydrolysis requires about 288.19 days (9.3 months).

Conclusion

FTIR characterization showed no significant differences among the products synthesized in this study. XRD analysis revealed an identical structure but differences in crystallinity. Both DFTUH-SD and DFTUAc-SD have micrometric particle size, irregular surface, and mesoporosity. These results confirm that DFTU formation is independent not only of the starting molar ratio of urea to furfural but also of whether distilled or non-distilled furfural is used. The in vitro hydrolysis rate depends on the synthesis method; DFTUAc-SD hydrolyzes 1.7 times faster than DFTUH-SD. Based on the hydrolysis rate of DFTUH-D, the product is expected to release urea and furfural for over 9 months in vitro.

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Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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