The Compostability of Denim Fabrics Dyed with Various Indigos

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Denim fabric samples representing current indigo dye sources and fabric structures were biodegraded in feedstock including food waste, manure, and animal bedding, which are typically composted at the Cornell Farm Services Composting Facility and processed under laboratory conditions for 77 days. Indigo types including dry denim, pre-reduced, and natural did not inhibit degradation as compared to undyed 100% cotton fabric. Additionally, fabrics tested as received from the mill and those tested post scouring degraded effectively. As expected, denim containing 24% polyester and 2% spandex retained overall fabric structure despite degradation of the cotton portion of the yarns.

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INTRODUCTION

With increasing concern about the sustainability of textiles and clothing, disposal, and end of life fate of fabrics and apparel have come under scrutiny. Indeed, the fate of waste textiles and clothing has increased in importance due to the rise of fast fashion and the corresponding increase in mass production of apparel and other fashion items. Fast fashion can be described as rapidly produced, low cost, and low quality apparel designed to be consumed and discarded quickly in response to short-lived trends. This in turn generates copious amounts of textile waste per annum as the fashion cycle has been severely shortened (Bhardwaj 2010; Niinimäki *et al.* 2020; Cuiffo *et al.* 2021). Among the range of potential solutions including recycling (Singhal *et al.* 2023), upcycling, incinerating, and reselling (thrifting), the possibility of composting and biodegradation for textiles comprised of natural fibers including cotton is an attractive alternative. Cotton is one of the most widely used fibers all over the world. It has found applications in all categories of textiles, most notably the clothing sector, because of its unique physical and chemical properties including versatility, hydrophilicity, durability, and softness (Krifa and Stewart Stevens 2016). The sustainability of the production and use of cotton and the resultant effects on land use, soil erosion, irrigation water, energy use, and greenhouse gas emission has been examined over the past few decades, all in a bid to produce and use cotton more sustainably (Daystar *et al.* 2017; Esteve-Turrillas and de la Guardia 2017;

Delate *et al.* 2021). For cotton, composting of textiles to be incorporated into soils for cotton growth could create a truly circular system.

In 2010, this research team published its first paper on cotton textile biodegradation (Li *et al.* 2010), combining both laboratory and compost facility data. Citations of that study are escalating and include studies of degradation of other fabric systems and in various environmental systems including aquatic (Zambrano *et al.* 2020, 2021) and simulated landfill (Sui *et al.* 2022). Our further studies have compared the effects of a wide range of cotton dyes and finishes on degradation behavior and partnered with experts measuring residual chemicals in soils post degradation (Sultana *et al.* 2019; Sui *et al.* 2020; Smith *et al.* 2021).

Simultaneously, significant efforts have been made to improve all aspects of cotton production from farming (Barnes *et al.* 2020) to fabric. Recent efforts have transformed the indigo dyeing processes used for manufacture of blue fabrics, most prominently denim, and mitigate impacts of the dyeing process on the environment (Periyasamy and Periyasami 2023). The use of pre-reduced indigo (Blackburn *et al.* 2009), ‘dry’ foam application of reduced indigo (Doudou *et al.* 2022), and natural indigo harvested from plants have been explored in efforts to reduce the environmental footprint of the indigo dyeing process. These alternatives to synthetic powdered indigo are particularly targeted towards reducing overall water usage in the dyeing process, decreasing associated wastewater, and reducing the reliance on petrochemicals for synthesis.

With excellent durability and unparalleled versatility, denim is ubiquitous as both a fashion and workwear fabric (Gokarneshan *et al.* 2017). Forecasts predict that the global denim market will reach $95 billion by 2030 (Smith 2023). While historically denim was produced from 100% cotton yarns, many current denim products include blended cotton, polyester, and elastane yarns (Shikha Sarker *et al.* 2016; Gokarneshan *et al.* 2017; Yusuf Daşan 2020; Akter *et al.* 2021), further complicating efforts to recycle or compost this fabric.

Currently, disposal of fabrics including denim as both post-industrial scrap and post-consumer clothing is straining waste management infrastructure (DeVoy *et al.* 2021). Potential solutions include re-using, recycling, and finally composting. Denim garment and textile use and reuse can be maximized *via* second-hand garment systems (Fortuna and Diyamandoglu 2017; Rynk and Ziegenbein 2022) and innovations in textile recycling (Dahlbo *et al.* 2017; McCauley and Jestratijevic 2023; Wagaw and Babu 2023). With landfilling and incineration of both unsold and post-consumer textile products recently banned in a growing list of places (Protection 2022; Reuters 2023), a final end of use solution for cotton textiles could include biodegradation (Mazibuko *et al.* 2019) in composting facilities. Other studies have shown that cotton textiles, cellulose, and cellulose derivatives are biodegradable (Sannino *et al.* 2009; Yaradoddi *et al.* 2020; Tian *et al.* 2022). There have also been studies on the biodegradability of composites incorporating denim waste (Haque and Naebe 2022).

Municipal composting has emerged as an effective way to recycle a variety of organic wastes into nutrient rich soil. Large-scale composting facilities operate at elevated temperatures up to 60 °C and both with and without turning to ensure oxygenation and microbial activity. Composting, therefore, provides an avenue to completely break down and recycle a wide range of materials including cotton fabrics such as denim (Ghosh and Jones 2021; Heisey *et al.* 2022; Sánchez 2022).

To assess the viability of increasing circularity of denim fabrics, the impacts of fiber composition and dye type on degradation need to be assessed. In the current study, 13 denim samples (Table 1) dyed using various indigo types and methods including an undyed control fabric and 3 commercially sold denim jeans samples were tested for biodegradation under laboratory conditions that mimicked the composting process for 77 days according to ASTM D5338 (2021). Denim fabric samples representing current indigo dye sources and fabric structures were biodegraded under laboratory conditions for 77 days in a medium of food waste, manure, and animal bedding, which was obtained from the Cornell Composting Facility. Indigo types including dry denim, pre-reduced, and natural indigo did not inhibit degradation as compared to undyed 100% cotton fabric. Additionally, fabrics tested as received from the mill and those tested post scouring degraded effectively. As expected, denim containing 24% polyester and 2% spandex retained overall fabric structure despite degradation of the cotton portion of the yarns. Simultaneously, these samples were also studied in the Cornell Compost Facility under large-scale composting conditions (Schwarz *et al.* 2023). To minimize the noise from the background carbon dioxide in the compost, the incubation media was mixed with inert perlite, for a more accurate carbon dioxide measurement (Solaro *et al.* 1998; Chiellini and Corti 2003). The amount of degradation was quantified by tracking the carbon dioxide released over time. The mass loss, fabric morphology, and degradation media (DM) parameters, including pH and carbon to nitrogen (C:N) ratios, are also tracked to further describe the extent of degradation (Solaro *et al.* 1998; Chiellini *et al.* 1999; Chiellini and Corti 2003; Rizzarelli *et al.* 2015; Markoska *et al.* 2018; Cinelli *et al.* 2019; ASTM D5338 (2021), ASTM D6440 (2023); Schwarz *et al.* 2023)

In the concurrent study, carried out at the Cornell Composting facility, significantly higher levels of degradation were measured by mass loss and visual inspection. These observations are unsurprising because large-scale composting achieves thermophilic temperatures, approximately 60 °C, whereas the lab study was run at ambient conditions throughout. The composting trial also kept track of other compost parameters including moisture, pH, soluble salts, organic matter (OM), total nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), total carbon (TC), carbon to nitrogen ratio (C:N), metals, and per- and polyfluoroalkyl substances (PFAS) (ASTM D6440 2023).

EXPERIMENTAL

Materials

Hydrochloric acid solution (37%) was purchased from VWR (Bridgeport, CT, USA) and diluted to 1 M with deionized (DI) water. Potassium hydroxide (KOH) and phenolphthalein indicator were purchased from VWR (Bridgeport, CT, USA). All chemicals were used as received. The KOH solutions were prepared by dissolving the appropriate mass of KOH pellets in deionized water. The immature compost used consisted of 53% food scraps and 47% manure and bedding from the Cornell School of Veterinary Medicine (by tons, as received) at the Cornell University Compost Facility (CFC), Ithaca, NY. This material had been composting for approximately 2 months. Perlite was purchased from Agway, Ithaca, NY, USA.

The denim samples used in the study were supplied by Cotton Incorporated and used as received (Table 1). Each sample except R-W (Brand 2) was provided in both a raw and washed form. All denim samples received at Cotton Incorporated were loom-state, meaning that they still contained size applied to facilitate weaving. The “Raw” connotation refers to that loom-state denim and would represent cutting room waste from apparel production. The “Washed” denim has been through two processes. The first process was a desize bath with amylase enzymes and heat to remove starch and any polylactic acid size applied to protect the yarns through weaving and add stiffness to the fabric for cut and sew.

Table 1. Sample Summary

ID	Dye Process/Dye	Denim Type	Fabric Weight (g/m^2)	Carbon Content (wt %)
A-R	Dry Denim	Raw	373	44.10
B-W	Dry Denim	Washed	373	45.81
E-R	Standard Pre-reduced	Raw	407	39.42
F-W	Standard Pre-reduced	Washed	407	45.12
I-R	Natural Indigo	Raw	424	40.88
J-W	Natural Indigo	Washed	424	44.66
M-R	Brand 1 (2% natural rubber)	Raw	398	44.50
N-W	Brand 1(2% natural rubber)	Washed	398	45.61
R-W	Brand 2(74% cotton, 24% PET 2% spandex)	Washed	418	51.35
U-R	Undyed	Raw	373	44.59
V-W	Undyed	Washed	373	39.73
Y-R	Brand 3	Raw	458	44.97
Z-W	Brand 3	Washed	458	44.83

All samples are 100% cotton except M-R and N-W, which contain 2% natural rubber and R-W, which contains polyester (PET) and spandex as noted.

Following the desize step, the fabrics were processed with a neutral cellulase enzyme and tumbled with Tonello’s No Stone plates. This combination emulated a stone wash process to remove some indigo and some cotton fibers from the surface of the fabrics, giving them a slightly worn appearance. Carbon content was measured in the Cornell Nutrient Analysis Laboratory (CNAL) by combustion analysis. The higher carbon content of R-W is consistent with the higher carbon content of polyester polymer (≈ 60 wt%) compared to cotton (≈ 42 wt%).

Fiber Morphology Characterization

Photos and scanning electron microscopy (SEM) images of samples were taken before degradation and on the 50th and 77th day of the disintegration testing. The microstructure of the denim samples as degradation progressed was examined using scanning electron microscopy (Zeiss LEO 1550 FESEM) at an accelerating voltage of 2 kV. The samples were coated using Au-Pd prior to imaging. Camera photos of samples were also taken before composting and on the 50th and 77th day.

Degradation Medium (DM)

Two-month-old compost inoculum, comprising food waste, manure, and animal bedding was obtained from the Cornell Composting Facility and stored in an airtight bucket at room temperature for 12 days prior to use in testing. C:N ratios, pH, and loss on ignition (LOI) were measured at CNAL and found as 21, 7.05, and 50.1%, respectively. These parameters were remeasured on completion of the study. The DM was sieved to less than

8 mm and large stones and sticks were removed by hand. Moisture content was calculated as 62.4% from the weight loss after overnight drying in an oven at 105 °C.

Denim degradation

Figure 1 presents a schematic of the degradation experimental set up. The degradation experiment followed a modified version of ASTM D5338 (2021). Modifications included storing samples at ambient room temperature rather than elevated temperature and use of KOH rather than Barium Hydroxide in the CO_2 trap to avoid formation of carbonate precipitates (Solaro *et al.* 1998; Chiellini and Corti 2003). Perlite was added to the system to aid in moisture retention. Perlite is also commonly used in horticultural applications to provide aeration and moisture because it can hold 3 to 4 times its weight of water (Markoska *et al.* 2018).

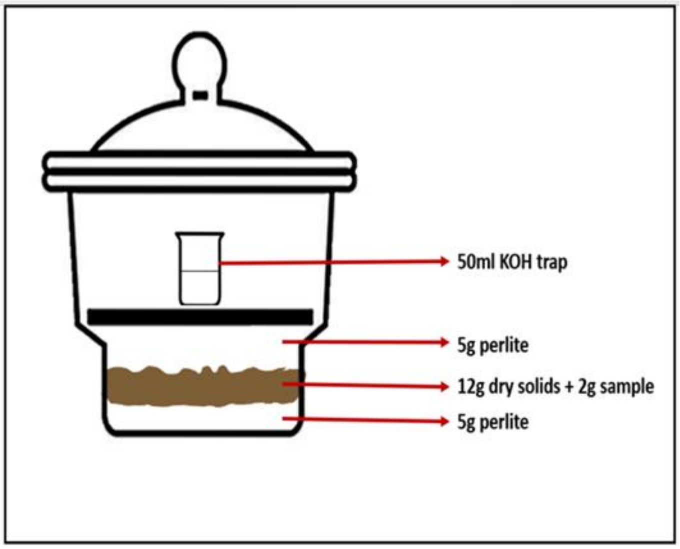

Fig. 1. Schematic diagram of desiccator set up for biodegradation experiments. DM, sample, and perlite are mixed together in the bottom of the desiccator. KOH solution is placed in the desiccator headspace to capture CO_2 produced.

Moist DM, containing 12 g dry solids, was mixed with 2.5 cm × 2.5 cm square denim swatches. The total weight of initial denim samples was 2 g, and the overall mass ratio was 1:6 (sample:DM). Sample:DM mixtures were placed in the bottom of a 2.4-L glass desiccator with 10 g of perlite added for moisture control. A total of 50 mL of KOH solution was placed in the top of the desiccator and served as a CO_2 trap. KOH solution concentration was varied between 0.1 and 1.0 M to match expected rate of CO_2 production as degradation progressed. All degradation tests were run in triplicate. Sampling included two controls. The raw undyed denim sample was included as a positive control to differentiate the effects of the different indigo dyeing techniques evaluated. Desiccators containing no denim samples were included as a blank/negative control to measure the CO_2 evolved by the DM alone and ambient CO_2. For all sample and control desiccators, DM was turned with a spatula at each sampling interval to ensure aeration of the DM. Lack of mold or fungus growth confirmed the system functioned in an aerobic rather than anaerobic mode.

CO_2 Production

CO_2 captured in the KOH trap was measured by titration with HCl. The ASTM 5338 (2021) method specifies monitoring CO_2 generation for 45 days. The authors allowed this study to run for an extra month to a total of 77 days. For the first 45 days, which is the period of most rapid degradation, titrations were performed every three days. Thereafter, titrations were performed weekly and finally biweekly until CO_2 production rate reached a steady state.

RESULTS AND DISCUSSION

All denim samples in this study were post-production but had never been worn. Different indigo dyes and dyeing methods were represented: Dry Denim (A-R, B-W) where a pre-reduced indigo is applied *via* foam, Standard Pre-reduced indigo (E-R, F-W), and natural indigo (I-R, J-W) both applied in a standard indigo dye range where the yarn was dipped into reduced indigo solution followed by an oxidation step. This was repeated several times to build up the desired color on the yarn. While the fabric weights varied from 373 to 458 g/m^2, all were within the expected range for denim jeans. All samples were comprised of 100% cotton yarns except M-R and N-W, which contained 2% natural rubber and R-W which included 24% polyester and 2% spandex. The dye types and methods used for samples M-R, N-W, R-W, Y-R, and Z-W were unknown. For all samples, R indicates that the samples were 'raw' and have not been scoured post-production while W indicates the samples were scoured at Cotton, Incorporated. Overall, these samples represented the range of denim jeans fabrics, current indigo dye chemistry and technology and typical denim on the market.

During the biodegradation experiment, carbon in the samples and DM was converted to CO_2. The CO_2 in the desiccator headspace was then captured in the KOH solution. The primary method for determining the extent of biodegradation in this study was assessment of the % of C in the initial sample that was converted to CO_2 in comparison to the blank negative control (DM only). The overall percent biodegradation is shown in Fig. 2. Early in the experiment, the control (DM only) produced more CO_2 than several samples containing denim. Because only control DM measurements were used in determining the percent biodegradation for all other samples, this resulted in an apparent negative degradation as the control generated more CO_2 than those samples. The relatively low CO_2 production by these denim samples was attributed to the relatively large size of denim pieces and slower colonization or rate of microbial activity at the fabric surface as degradation initiated. Over the course of this study, between 18% and 33% of the carbon contained in each sample was converted to CO_2. Data points shown are the averages of 3 samples with standard deviation bars omitted for readability. As expected for this type of system, variation among samples occurred. Some variation in the data was attributed to elevated temperatures in some samples due to interference from sunlight depending on the position of the desiccator and its proximity to the window. Because this affected the blanks as well, the effect was mitigated for the batches it affected.

Although differences in the mean % biodegradation are evident, relative standard deviations among data were large, in some cases approaching 5%. To assess whether any of the specific differences in samples including fabric weight, indigo dye type, washed *vs.* raw or fiber content resulted in a statistically different degradation behavior, paired t-tests at 95% confidence were conducted on the degradation data. Samples A-R, B-W, E-R, F-

W, and I-R, J-W allowed comparison of three types of indigo dye to be compared with the undyed control sample in both the raw and washed states (U-R, V-W). At 95% confidence using Microsoft 365 enterprise edition Excel (Microsoft Corp., Redmond, WA, USA), addition of indigo did not impact degradation in comparison to the undyed control. Most of the p values resulting from this pairwise t-test were well above 0.05, as shown in Table 2. Sample F-W *vs.* V-W p = 0.056, which while not meeting the test for significance, does indicate that F-W was degrading more slowly.

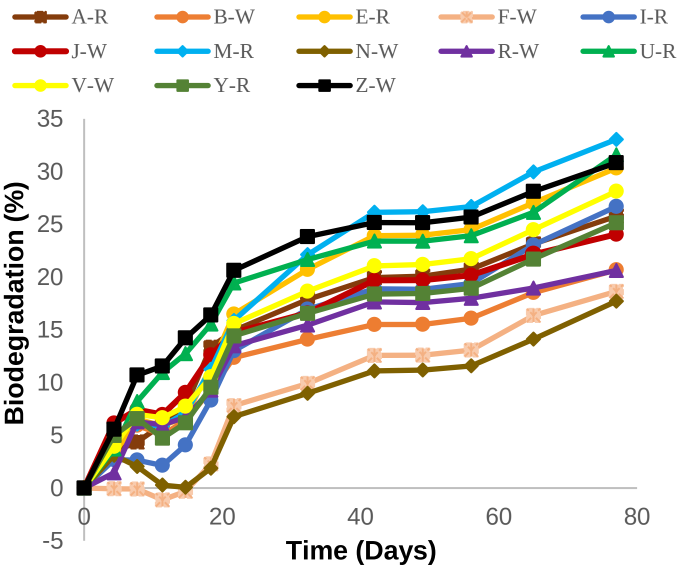

Fig. 2. Percent biodegradation determined as % C converted to CO_2 in comparison to DM only control

Likewise, a pairwise comparison of samples of each sample pair in the raw and washed sets yielded high p-values of over 0.05 for most pairs. Being raw *vs* washed, therefore, had no statistically significant effect on the rate or level of degradation of those samples, as shown in Table 2. Sample F-W again stood out as degrading more slowly than the raw counterpart E-R, and N-W also had a low p-value compared to M-R. Both of these samples also showed very low degradation in the second week of sampling. Potentially, this represents some shrinkage in the washing process, increasing fabric compactness and inhibiting microbial attack. In both cases, however, biodegradation continued throughout the study. A pairwise comparison based on sample fabric weights did not find any difference in the % biodegradation. Analysis comparing the heaviest and lightest fabrics is included in Table 2. Additionally, comparing samples with similar fabric weight, but different fiber composition showed no significant difference in biodegradation during the course of the experiment. This result reflects the structure of the yarns within the R-W sample, where all cotton is at the surface and available for biodegradation while polyester and spandex components are contained in the core.

The extent of biodegradation was also estimated by evaluating the fabric weight loss using Eq. 1. In the equation, W_t (%) is the percent of weight loss after t days of biodegradation, W_0 is the initial weight of the fabric in grams (g), and W_t is the weight of dry fabric after t days of biodegradation in grams (g), where t = 77 days. Mass loss was determined by collecting all remaining fabric and fiber material and removing DM from surfaces by brushing.

$$W_t(\%) = \frac{W_0 - W_t}{W_0} \times 100 \qquad (1)$$

Table 2. p-Values of Paired t-tests Performed on Sample Pairs Using Mean % Degradation Data at Final Data Point

Indigo Dyes *vs.* Undyed Control	
t-test	**p-value**
A-R vs U-R	0.158
E-R vs U-R	0.362
I-R vs U-R	0.25
B-W vs V-W	0.092
F-W vs V-W	0.056
J-W vs V-W	0.311
Raw *vs.* Washed	
t-test	**p-value**
A-R *vs* B-W	0.18
E-R *vs* F-W	0.01
I-R *vs* J-W	0.391
M-R *vs* N-W	0.052
U-R *vs* V-W	0.255
Y-R *vs* Z-W	0.127
Fabric Weight	
t-test	**p-value**
373 vs 458 g/m^2	0.274
Fiber Content	
t-test	**p-value**
J-W vs R-W	0.301

The results for the various samples are as shown with an average value of 49%, a high of 62% and a low of 27%. This data are compared with the data obtained from carbon dioxide evolution in Fig. 3. In all cases, the calculation of degradation by % of mass loss was greater than calculated by CO_2 production. Some over-estimation of mass loss resulted from the difficulty in retrieving all remaining samples from the DM after 77 days. Sample R-W, which contains polyester and spandex as well as cotton, had the closest agreement between the two measurements. Because the polyester and spandex had not degraded, much of this sample remained intact and easy to retrieve. All other samples were significantly disintegrated and difficult to separate from DM. R-W also had the lowest degradation, measured by mass loss, of all samples.

Fabrics were sampled at 0, 50, and 77 days to monitor the extent of degradation macroscopically and microscopically, as shown in Fig. 4. At 50 days, fabric samples were visibly degraded. The SEM images confirm that yarn twist was loosening, and fiber breakage and fibrillation had started. After 77 days, fabric samples were disintegrating and had visible holes and frayed edges. As mentioned above, camera images confirmed that samples other than R-W, which contains synthetic fibers, were fragmenting into smaller pieces and were no longer intact. In SEM images, many samples had a film, potentially representing microbial activity, covering the cotton fibers. Figure 5 clearly shows the undegraded polyester fibers (circled) and highly degraded cotton fibers in sample R-W. The cotton portion of the yarns was readily exposed to the DM and biodegrading similarly to the 100% cotton samples.

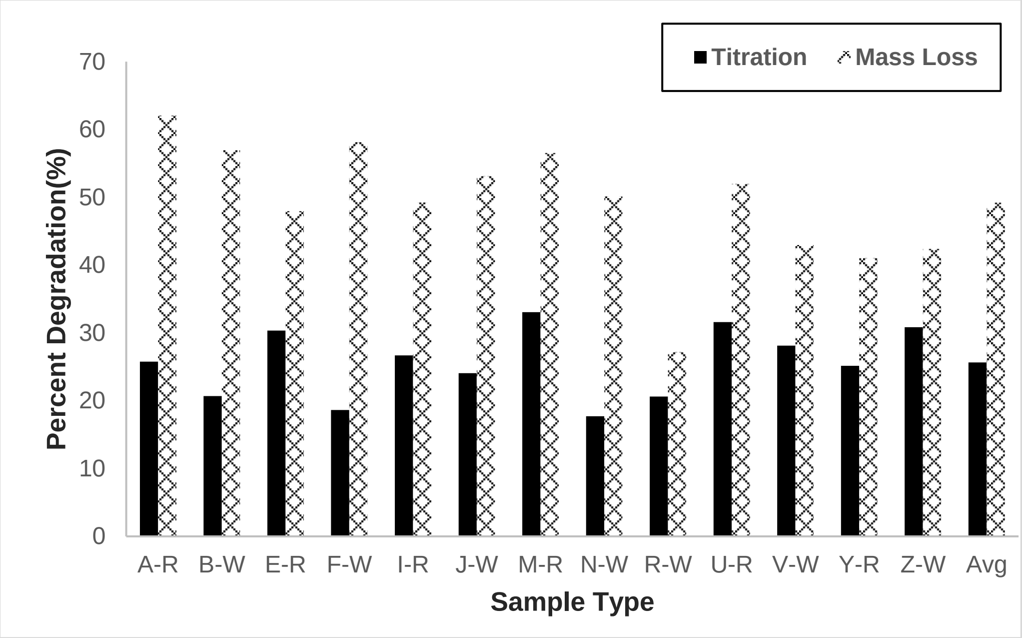

Fig. 3. Comparison between percent degradation determined by biodegradation (conversion of C to CO_2) and mass loss

Previous reports considered different cotton fabric constructions and finishes in different degradation media including natural soil and water. Smith *et al.* (2021) reported lower sample % weight loss in the same time period as the current study, likely resulting from less active degradation media. The SEM images did not show evidence of microbial growth on the textile surfaces.

Notably, this study used significantly higher sample to soil mass ratios than the current study (Smith *et al.* 2021). Li *et al.* (2010) also reported lower % weight loss for cotton knit samples in natural soil, however, with a lower sample to DM ratio evidence of microbial activity. In these systems and reports on degradation of cotton microfibers in aqueous medium, dyes or finishes present did affect the degradation rate, although all samples ultimately degraded. As discussed above, variations in indigo dye type or washing condition had minimal impact on degradation.

The DM also changed over the course of the study. Initially, the DM was brown with a pungent odor. Over the course of the degradation experiment, this odor dissipated. While the pH of the initial DM was 7.05, DM in all 42 desiccators increased in pH to an average of 8.47, as shown in Fig. 6. The increase in pH is consistent with results reported in the composting facility study on these same materials (Schwarz *et al.* 2023) and is typical of the composting process (Rynk and Ziegenbein 2022). The C:N ratio of the DM decreased from the initial value of 21 to an average of 16.9 (Fig. 7) over the course of the study. This decrease confirms that carbon from the DM was also being converted to CO_2, as expected.

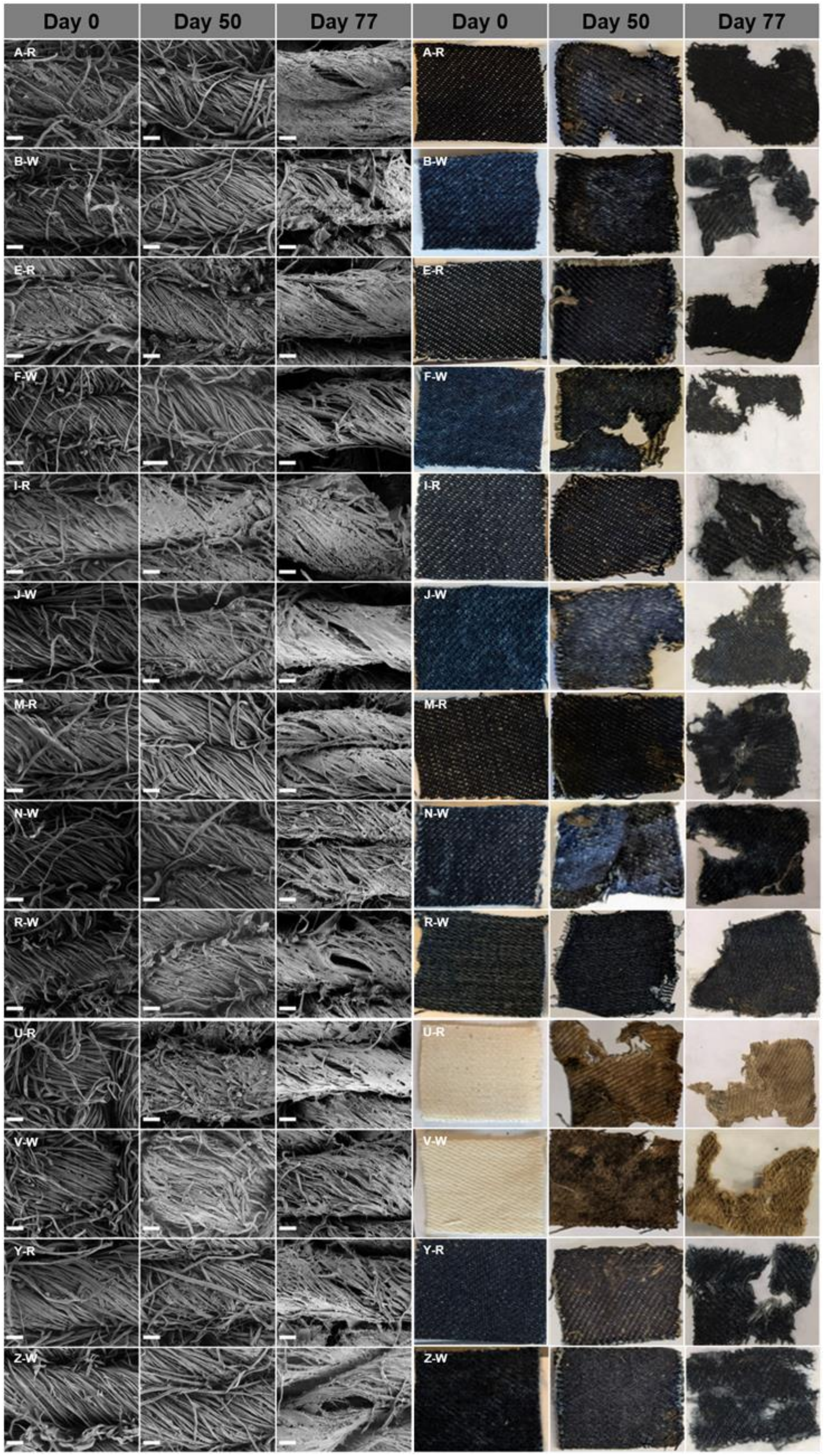

Fig. 4. Camera and SEM images of samples as received and after 50 and 77 days of biodegradation

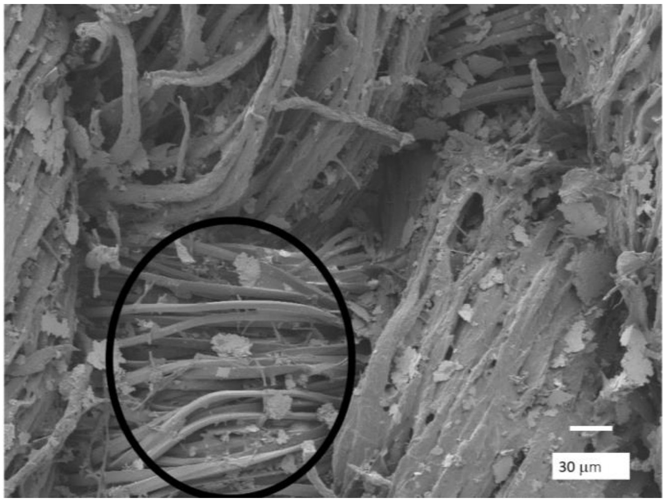

Fig. 5. Higher magnification image of sample R-W after 77 days. Undegraded polyester fibers circled.

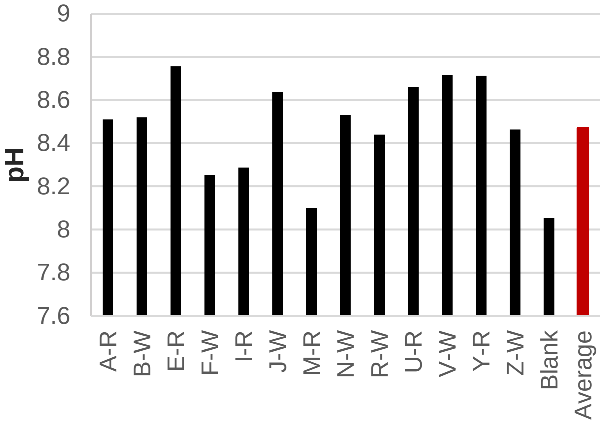

Fig. 6. pH of DM at conclusion of the biodegradation experiment. Red line represents the starting pH

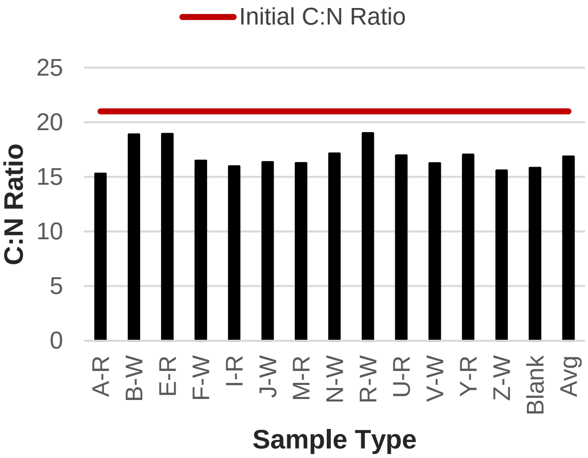

Fig. 7. C:N ratio for DM at conclusion of composting experiment. Red line represents the starting C/N ratio

CONCLUSIONS

1. Overall, all 100% cotton denim samples degraded similarly, regardless of fabric weight, washing, or indigo dyeing process, and all could be expected to behave similarly in composting conditions.
2. All samples showed significant degradation over the course of the experiment, as evidenced by photographs and SEM images. However, both CO_2 evolution [Min: 18%, Max: 33%, Ave: 26%] and mass loss [Min: 27%, Max: 62%, Ave: 49%] results indicated that degradation of samples was not complete at 77 days.
3. Stability of the sample containing 24% polyester and 2% spandex was evident in low mass loss and cohesiveness of the fabric despite significant degradation of the cotton

portion of the fabric. The SEM images confirmed that the cotton portion of the fabric was degrading, while the polyester, unsurprisingly, was not.

4. Change in the pH and C:N ratios of the DM were tracked before and after composting. The former increased while the latter decreased from its initial value, indicating that the DM was changing as expected and remained active as the experiment progressed.

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