MRS Communications (2021) 11:662–668

https://doi.org/10.1557/s43579-021-00094-0

MRS Communications

Research Letter

A study of formability of palm leaf materials using Limiting Dome Height testing

Debapriya Pinaki Mohanty, and **Anirudh Udupa,** Center for Materials Processing and Tribology, Purdue University, West Lafayette, IN 47906, USA
A. R. Anilchandra, Department of Mechanical Engineering, BMS College of Engineering, Bengaluru 560019, India
James B. Mann, M4 Sciences Corporation, Lafayette, IN 47901, USA
Srinivasan Chandrasekar, Center for Materials Processing and Tribology, Purdue University, West Lafayette, IN 47906, USA

Address all correspondence to Debapriya Pinaki Mohanty at **dmohant@purdue.edu**

(Received 24 June 2021; accepted 3 September 2021; published online: 16 September 2021)

Abstract
Deformation response of *Areca catechu* palm leaf-sheath under biaxial stretching is evaluated using Limiting Dome Height (LDH) testing. It is shown that the leaf material has high formability, with biaxial limit strains ~ 0.4, comparable to that of ductile sheet metals. Hydration of the leaf-sheath prior to the forming increases the limit strains by approximately 500% and lowers deformation forces by up to 85%. The potential of the LDH test for characterizing the stretch-forming response of plant materials is also demonstrated. Implications for single-step forming of plant leaf materials into products and manufacturing of eco-friendly foodware are discussed.

Introduction
The foodware segment is perhaps the largest user of plastics. Much of this is in single-use applications such as utensils (e.g., plates, bowls) and packaging. Given the deleterious effects of plastics on the environment,[1] there is much effort ongoing to develop environmentally sustainable material alternatives for these single-use applications. In recent years, plant-based materials processing has emerged as an attractive option, notably foodware made by molding of pulped wood fiber or bagasse. This production route, wherein plants serve as the main source of the fibrous materials, has many attractive features related to throughput, cost and scalability. But these benefits are somewhat offset by the intermediate, highly resource-intensive pulping and drying steps involved in the processing. The pulp-based processing also often uses fillers and chemicals (~ 30%).

A more attractive manufacturing route from a sustainability standpoint is production of foodware utensils, such as plates, cups and bowls, in a single-step, by direct deformation processing (forming) of plant materials.[2] It is in this context that—*Areca catechu*–a palm variety cultivated for nearly 2000 years in India and South-East Asia[3,4] and the subject of this study, stands out (Fig. 1). Leaf sheath from this tree, typically a few millimeters thick and with a hierarchical structure composed of fibers, matrix and porosity, can be directly formed into plates and bowls in a single step. The large shape changes are accomplished by stretch forming of hydrated sheath material between dies, analogous to forming of sheet metal. Various foodware products produced from areca palm leaf-sheath are shown in Fig. 1, along with relevant structural attributes. The single-step forming avoids use of fillers and additives, and intermediate processes such as pulping. And the sheath material biodegrades in ~ 100 days compared to hundreds of years for plastics.[5] Since the palm is grown mainly for its nuts, the sheath, that is seasonally shed by the tree, is a waste product that is put to use, highlighting another attractive advantage of the processing.

Areca-leaf foodware manufacturing today is carried out in small-scale shops—cottage industries—and is mainly confined to the emerging and developing economies of Asia. The foodware is attracting increasing attention across the world because of its sustainability attributes, complemented by esthetic qualities. The palm leaf product manufacturers have generally relied on empiricism and intuition of material behavior for their product design, development and manufacturing. Discussions with manufacturers indicate that the demand for the products is far in excess of supply, primarily due to raw material (leaf) and processing constraints.

Given the intriguing formability characteristic of the leaf material, especially its capacity to undergo large shape changes, that has long been known empirically to practitioners, we recently embarked on a program to understand the mechanical behavior of these leaf materials and associated structural basis. The initial results from this program[6] showed that the leaf-sheath has a hierarchical microstructure broadly similar to other palm leaf materials, and woods. Importantly, uniform elongations of as much as 35% in uniaxial tension, and (sheath) thickness reductions of ~ 70% in compression and rolling, prior to failure, were demonstrated under suitable hydration conditions.

The present study builds on the prior results pertaining to mechanical response and explores formability characteristics of the leaf material using Limiting Dome Height (LDH) testing [Fig. 2(a)]. The LDH is a classical test that has been widely used in the sheet metal forming sector to assess formability. It also closely mimics the punch stretch-forming process used to manufacture areca foodware. Our LDH test results confirm

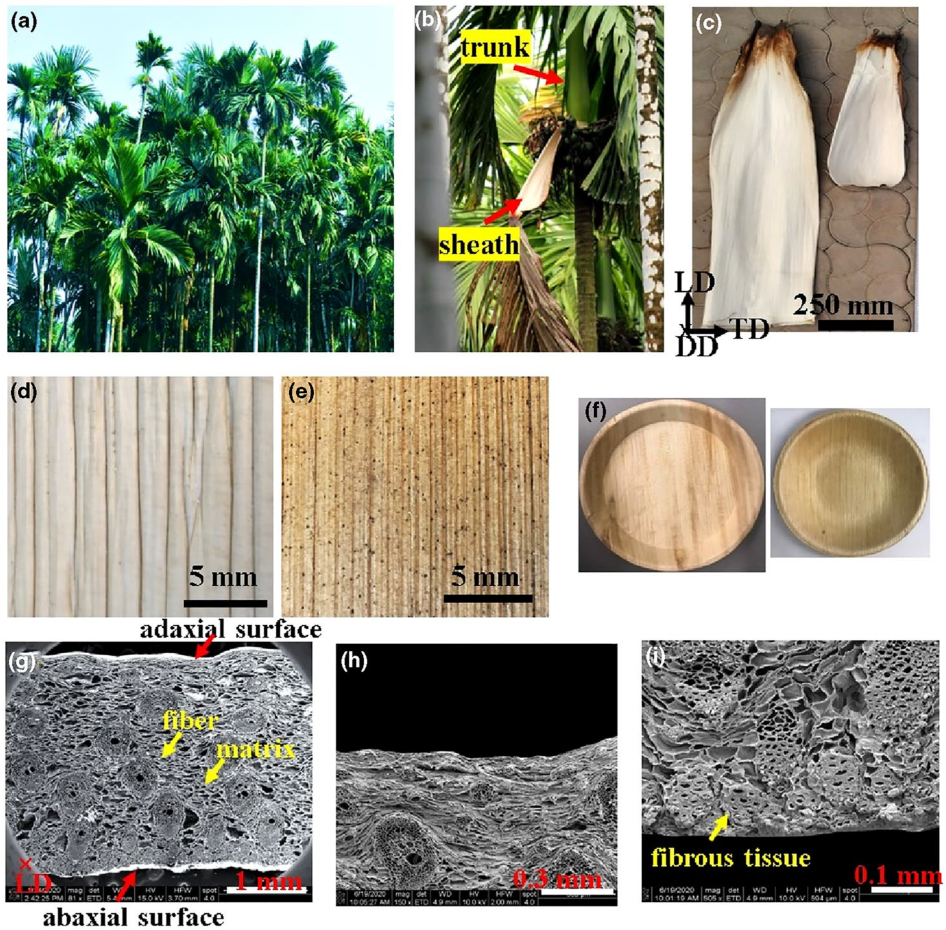

Figure 1. Overview of areca catechu palm and palm leaf foodware products (a) Areca palm trees in a plantation in the Western Ghats region in South India. (b) Close-up picture of a tree showing leaf-sheath. (c) Raw leaf-sheath. The orientation nomenclature is length/longitudinal direction (LD), depth/thickness direction (DD) and transverse direction (TD). The two surfaces of the sheath are (d) adaxial surface (inner) and (e) abaxial surface (outer). Note the difference in surface texture between the two surfaces. The striations on the surfaces run parallel to LD. (f) Formed foodware (left) plate and (right) bowl. SEM images of microstructure (g) Sheath cross-section in plane perpendicular to LD. (h) and (i) Higher magnification images of the cross section in adaxial and abaxial surface regions, respectively.

the anecdotally known large formability of this leaf material and, importantly, quantify this formability in a deformation processing framework. Parameter effects such as arising from hydration and step-wise loading are studied in the context of optimizing the areca forming process. The results are a key step towards establishing forming limit diagrams (FLDs) for these materials and the structural basis for their high formability. They should enable improved systematic design of product shapes that can be manufactured from the leaf materials, and forming process optimization to maximize shape changes for high-aspect ratio foodware products.

Background

Figure 1(a) shows areca palm trees in their natural setting in a plantation in the mountainous Western Ghat region of India. The sheaths are sheet-like structures, with physical attributes resembling a leaf-wood hybrid, that are connected to the main stem or trunk of the tree [Fig. 1(b) and (c)]. Typical sheath dimensions are length, 0.25 to 1 m; width, 0.1 to 0.5 m; and thickness of 3 to 4 mm. The density of the sheath material is 0.4 ± 0.1 g/cc. The sheaths serve to protect inflorescence during the early stage of flower development but are shed (seasonally) in the later stage; each tree typically produces ~ 10 sheaths every season. The sheath can therefore be termed as a “waste product” with the nut being the primary commercial product. It is this sheath that is formed by a punching or stretch-type forming process, after a hydration treatment, into plates and bowls. The commercial stretch forming process closely resembles the LDH configuration of Fig. 2(a), except that in the commercial process, the sheath is pressed against a second lower die placed underneath the sheath that confines the deformation. Examples of commercially available plates and bowls, formed in this manner, are shown in Fig. 1(f).

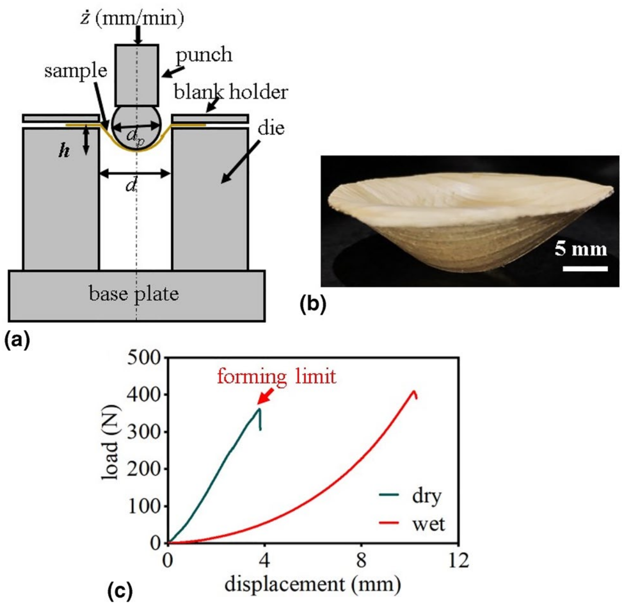

Figure 2. Limiting Dome Height (LDH) test and test output (a) Schematic of experimental setup used in the LDH test for biaxial stretching. (b) Cup formed by the stretch-forming. (c) Load vs displacement curves for dry and hydrated (wet) sheaths. Punch velocity ($\dot{z}$) is 5 mm/min.

It is useful at this stage to define some attributes of the sheath and related nomenclature. The broad inner face of the sheath that faces the palm tree stem is called the adaxial surface [Fig. 1(d)]; this surface usually forms the inner side of the foodware product. The opposite outer face of the sheath is the abaxial surface [Fig. 1(e)] and constitutes the outer side of the product. The abaxial side is much darker visually than the adaxial side, besides being stiffer and stronger. Both surfaces have striations that run along the length direction of the sheath. These striations are the principal source of surface roughness, both in the sheath and formed product. On the adaxial surface of a raw sheath, the striations have a peak-to-valley height of ~ 400 µm, and lateral spacing of ~ 3 mm; while on the abaxial surface, the striations are much smaller in height and more closely spaced, with peak-to-valley height of ~ 50 µm and lateral spacing of ~ 0.5 mm. The directions/orientations of relevance in the sheath are labeled in Fig. 1(c)—length/longitudinal direction (LD), parallel to the direction of the striations; depth/thickness direction (DD), along the sheath thickness; and the third, transverse direction (TD), that is perpendicular to the striations in the plane of the sheath.

An overview of the sheath microstructure is provided in the SEM images in bottom row of Fig. 1(g–i). The sheath interior has a hierarchical microstructure composed of fibers (27 vol%), with effective diameter, 200 to 500 µm, and length > 50 mm, that are embedded in a matrix. The fiber lengths are oriented parallel to LD. Both the fibers and matrix contain porosity, with the porosity volume being ~ 60%. A thin, denser layer—the epidermis—of thickness < 50 µm, adjoins the adaxial and abaxial surfaces. The epidermis comprises thick-walled cells of low porosity and lignified, resulting in a rigid and scarified layer. The epidermis provides additional strength and stiffness to the sheath, analogous to a composite sandwich panel.[7] In addition, clusters of small fibrous tissue (diameter ~ 50–100 µm) are present on the abaxial surface, see Fig. 1(i), likely increasing the stiffness/strength of the material in this region but lowering its ductility. This fibrous tissue is however absent on the adaxial surface [Fig. 1(h)]. The adaxial surface is hence likely to be more flexible, as well as more ductile, compared to the abaxial surface. Further details about the microstructure, physical attributes and mechanical response of the sheath can be found in Refs. 6–8. This hierarchical sheath microstructure is broadly similar to that of various palm leaf materials and woods.[8]

Experimental

Raw areca sheaths for the LDH experiments were obtained from a plantation in Tumkur near Bangalore, India. The sheaths were from a group collected during the 2019 summer season. The sheaths after collection had been cleaned using dry compressed air and stored inside plastic zipper bags, to avoid any contact with atmospheric moisture and prevent growth of fungus. The formability of the sheath was characterized by measuring forming limit, i.e., punch penetration to failure, in a LDH test, see Fig. 2(a). The LDH test samples were of size 40 mm × 40 mm × 3 mm (thickness) that were cut out of the sheaths just prior to the experiments.

Limiting Dome Height (LDH) test

The LDH test is one of the most widely used methods for characterizing formability of sheet metals.[9,10] In this test, a thin sheet of material, the test sample, is clamped along its periphery and then axi-symetrically stretched (expanded) into a cup/bowl form, see Fig. 2(a) and (b). The stretching is accomplished by application of a hemispherical punch that is pressed onto the unclamped central (disk-shaped) region of the sample; this loading is commonly described as one of biaxial stretching. The LDH test configuration of Fig. 2(a) closely resembles the actual sheath forming process[2] that is used to make foodware such as cups, plates and bowls, except that there is no bottom die in the LDH.

An MTS tensile tester (maximum load—2 kN) was adapted to carry out the LDH test. The hemispherical punch of diameter, d_p = 25 mm, was held in the movable upper jaw of the tensile test machine, while the lower jaw was replaced with the LDH die with hole diameter (d) of 30 mm. The sheath test sample was clamped along a circular peripheral region using a blank holder and formed into a cup by the punch-loading in displacement control mode [Fig. 2(a) and (b)]. The diameter of the unclamped sample central region was equal to the diameter of the hole in the die but slightly larger than the punch diameter, as is typical of LDH testing. The sheath that forms the inner side of the cup is thus in contact with the punch, while the other side is free. A thin Teflon film (solid lubricant) was placed between the sheath and punch surface to reduce the interfacial friction.

In a typical test, the punch moved downward at a velocity of $\dot{z}$ mm/minute until the sample failed. The load usually increased with displacement over much of the test duration up and until onset of sample failure at a maximum penetration depth (h). At this point, a sharp load decrease was usually noted [Fig. 2(c)]. In contrast to the areca forming process, which uses heated dies to prevent sample spring-back and lock in the sample shape,[2] the punch in the LDH test was kept at room temperature. Spring-back of the sample was prevented by flowing hot air at 120°C for 3 min, at the maximum load, before releasing the load.

The formability is reported and assessed in terms of the maximum (limit) strain developed at the sample pole (bottom-most point of cup), at failure. Based on assumptions of zero friction at the interface between the punch and sheath, and isotropic material response, the maximum in-plane strain (ϵ) at the pole is obtained using a solution for this loading case provided by Hill,[10,11] as $\epsilon = \ln\left(1 + (2\mathrm{h}/\mathrm{d})^2\right)$. Note that this is equal to one-half of the through-thickness strain. Here, h is the maximum punch penetration depth, and d is the diameter of the bowl corresponding to its periphery at this limiting depth. Both d and h were measured under load in the LDH tests for each sample. It is this pole strain that is reported as the forming limit strain in the ensuing.

Parametric effects

Several parameters, of potential relevance to areca leaf formability, were varied in a series of LDH tests. These parameters and associated test conditions are listed and described below. Unless otherwise noted, the LDH tests were done with a punch velocity ($\dot{z}$) of 5 mm/min, monotonic continuous loading, and with hydrated (wet) sheath samples. The hydration treatment was a 2-h immersion in water just prior to the test.

- Forming of areca foodware is typically carried out by applying the punch-load to the adaxial surface. Hence, the adaxial side becomes the inner surface of the formed product, i.e., the surface in contact with the food. Since the adaxial and abaxial surfaces have different structural and physical attributes,[6,8] see also Fig. 1, the LDH tests were conducted with both adaxial and abaxial sides loaded against the punch to determine differences, if any, in the formability.
- The effect of hydration was evaluated by testing dry sheath samples, i.e., without hydration, and comparing the measured forming limits against those of hydrated samples.
- The effect of loading (deformation) rate was studied using punch velocity $\dot{z}$ of 1, 5, 10 and 20 mm/min.
- The effect of stepwise loading was studied in a set of experiments wherein the punch was loaded in incremental displacement steps of 3 mm up to the sample failure. Furthermore, at the end of each 3-mm step, the displacement was held fixed for durations of 15 s, 30 s and 60 s.

Results

Figure 2(b) shows a cup, with aspect ratio h/d of 0.33, produced by the biaxial stretching in LDH test. This cup is analogous to a bowl produced by a commercial (small-scale) areca sheath forming process. But its aspect ratio is higher than that of commercial bowl products ($h/d < 0.2$). Interestingly, dimension/shape measurements showed the cup in Fig. 2(b) to closely resemble a hemispherical cap, except in the very vicinity of the clamped sample periphery, even though a lower die was not used to confine the deformation and constrain the shape change as in the commercial forming process.

Typical load–displacement to failure relationship obtained for the hydrated and dry samples are shown in Fig. 2(c). The failure in both cases always occurred at the pole of the cup. The penetration depths at failure (h) in the dry and hydrated conditions are seen to be 3.8 mm ($h/d = 0.13$) and 10.1 mm ($h/d = 0.34$), respectively, corresponding to maximum (pole) strains of 0.06 and 0.38 in the corresponding sheath samples. For reference, the maximum strain in a commercial bowl product is ~ 0.15. It is clear from the load-penetration curves and the strain values that the hydration treatment produces a significant increase in the formability and forming limit. Similar large increases in the strain at failure with hydrated samples have also been observed in uniaxial tensile testing of this sheath material.[7] Figure 2(c) also shows that the force required to produce a given shape change or punch penetration is significantly lowered by the hydration. For example, the maximum load, occurring almost at the failure limit, with the dry sample is ~ 350 N; whereas the corresponding load at the same penetration depth of 3.8 mm with the hydrated sample is < 50 N, an ~ 85% load decrease. Thus, both from a shape change standpoint and force/energy considerations, the hydration treatment has a remarkably beneficial effect on formability.

Figure 3 summarizes the key formability results from the parametric studies involving loading rate and stepwise loading. The top row in Fig. 3 shows the load–displacement curve and strain data for the various loading rates. The pole strain at failure is seen to decrease with an increase in the loading rate, from ~ 0.38, at the smaller loading rates of 1 and 5 mm/min, to ~ 0.25, at the highest loading rate of 20 mm/min. This represents a significant decrease (~ 30%) in formability. Concomitantly, there is a small increase in the punch (deformation) force. These results are somewhat analogous to the effects of strain rate on ductility and (yield) strength of metals. Note also that at the two smaller deformation rates, the load–displacement curves in Fig. 3 (top left) essentially overlap one another.

The effects of stepwise loading on the load–displacement curve and forming limit strain are depicted in the bottom row

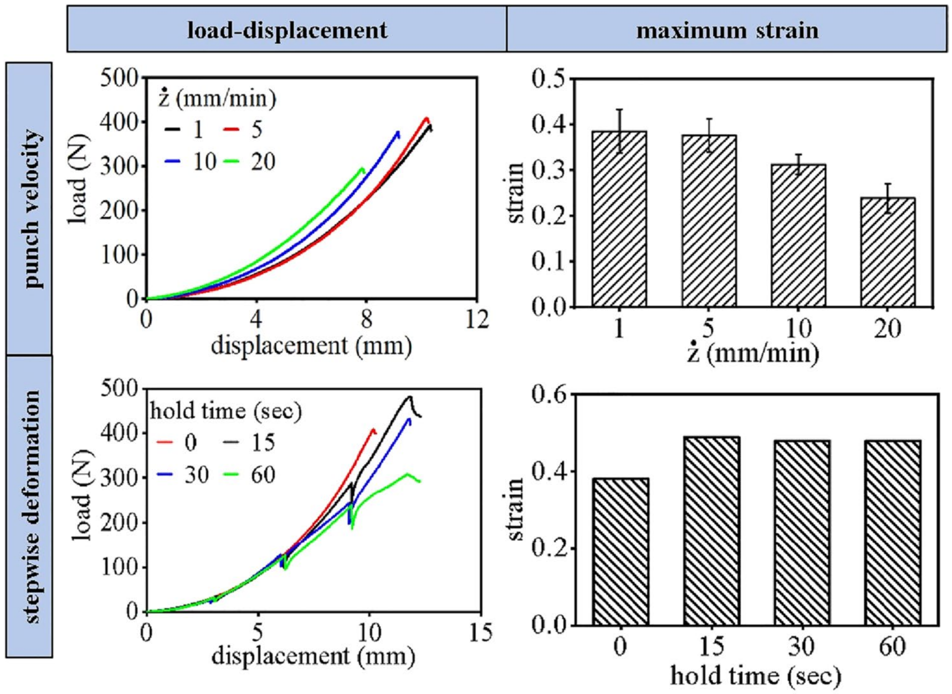

Figure 3. Load–displacement curves and forming limit strains. (Top row) Effect of punch velocity ($\dot{z}$). (Bottom row) Effect of imposing stepwise deformation.

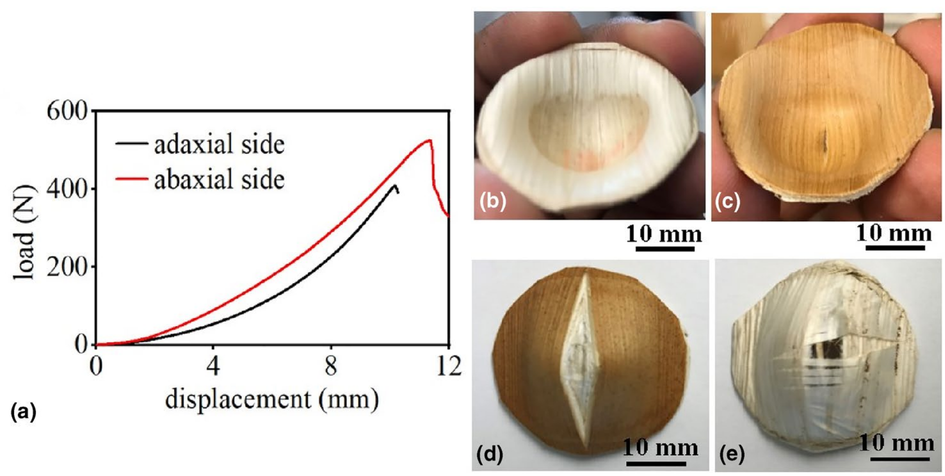

Figure 4. Deformation response of sheath for adaxial and abaxial surface loading. (a) Load–displacement curves. (b) and (c) Inner surface of the bowl when load is applied to (b) adaxial and (c) abaxial surface. (d) and (e) Failure region on the bowl outer (d) abaxial surface and (e) adaxial surface. Failure in both loading cases occurs at the pole. Note, however, the difference in failure morphology.

of Fig. 3. There is an ~30% increase in the limit strain when the loading is imposed under stepwise displacement control compared to the monotonic continuous loading case (hold time = 0 s, Fig. 3 bottom left). This increase in failure strain is seen to be independent of the hold time duration. The effect of stepwise loading on the deformation force is negligibly small in the initial stages of loading, up and until 6 mm of punch penetration. Beyond this point, and until sample failure, the deformation force is seen to be smaller for the larger hold times.

Figure 4 encapsulates the formability results for punch-load application to the adaxial and abaxial surfaces. The former corresponds to the loading configuration used in the commercial areca-forming process. The load–displacement curves [Fig. 4(a)] show that the penetration depth at failure is somewhat larger when the load is applied to the abaxial surface ($h = 11.8$ mm) than the adaxial surface ($h = 10.1$ mm). This is reflected in the higher strain at failure in the abaxially loaded sample ($\epsilon \sim 0.48$) compared to the adaxially loaded case

($\epsilon \sim 0.38$). The larger strain at failure in the abaxially loaded sample is likely due to the outer cup surface, the adaxial surface, being more ductile, due to its structural characteristics, as noted earlier.

Figure 4(b)–(e) show macrographs of the formed products and the failure regions for the two loading cases. The inner surfaces of the formed products are shown in Fig. 4(b) (adaxial loading) and Fig. 4(c) (abaxial loading). The surfaces are smooth in both cases, but with pronounced differences in the surface texture and shading. The latter characteristics reflect the inherent differences in the physical attributes of the sheath adaxial and abaxial surfaces. The region of punch load application, that is the contact region between the punch and the sheath surface, is also well-demarcated in the figures as a contrast between the central region of the cup and the peripheral region. Besides the formability change, the other striking difference is in the failure mode, see Fig. 4(d) and (e). In both the loading cases, the failure is seen to occur on the outer surface of the cup and at the pole. For the adaxial loading, the failure occurs by "splitting" of the outer abaxial surface; this splitting involves displacement of material in a direction perpendicular to the fiber length orientation [Fig. 4(d)]. In the abaxial loading case, the failure on the outer adaxial surface involves tearing of the epidermis skin layer [Fig. 4(e)]. These types of outer surface failures were observed consistently across multiple specimens.

Discussion

The LDH tests have provided a first-of-a-kind, quantitative characterization of formability for areca palm leaf-sheath under stretching conditions that mimic the commercial sheath-forming process. The results have shown that the sheath material is quite formable under this type of loading, with forming limit strains as high as ~ 0.4. These strain values are similar to the limit strains reported for highly ductile and formable alloys such as Al and Cu (0.2 to 0.5).[12,13] It is also much greater than estimated strain values (0.15) in commercial products such as plates and bowls, suggesting possibilities for forming high-aspect ratio products such as deep cups and tumblers. Grid-deformation LDH experiments planned for the near-future will enable strain mapping across the sample surface and further refinement of the strain estimates.

Hydration treatment of the sheath prior to the LDH loading has a strong and beneficial effect on the formability, with the hydrated sheath limit strain being ~ 6× that of dry (non-hydrated) samples. Concomitantly, the deformation force to realize a given shape change is also lowered, by as much as 85%. Both of these beneficial effects of hydration, also observed in uniaxial tension/compression and rolling,[6] can be of value for expanding the scope of areca leaf products. They also suggest a general means for enhancing the formability of plant leaf materials. Other parameters that have been found to increase the forming limit strain, but to a smaller degree than hydration, are stepwise deformation and small loading rates.

The experiments have also shown that when the punch-loading is applied to the abaxial surface, a greater forming limit strain can be achieved compared to adaxial surface loading. The latter corresponds, however, to the loading configuration used in the commercial sheath-forming process. By turning the material inside out for forming, so to say, relative to the commercial process, the limit strain can be increased by ~ 25%, representing a significant improvement in formability. While there are factors likely related to diffusion, aesthetic features, and structural integrity, that make areca foodware manufacturers settle for the less formable adaxial loading configuration in practice, the results suggest that there are opportunities also for forming using the abaxial surface loading for special product cases. Potential examples of such cases include production of high-aspect ratio products and foodware with short lifetime requirements (< 45 min). Taken together, our observations pertaining to process and material parametric effects on formability can be integrated and used to optimize leaf-sheath forming processes, as well as extend their scope.

The study has shown that classical formability tests, used to evaluate sheet metal forming limits, can also be applied to study formability of plant leaf materials and woods in quantitative terms. This sets the stage for understanding the structural basis of high formability in select plant material systems. The availability of forming limit diagrams for plant materials, analogous to sheet metals, can help accelerate the development of new eco-friendly and sustainable products, besides enhancing current forming processes for plant materials. We plan to explore these aspects in the near future.

Conclusions

The deformation response of *areca catechu* palm leaf-sheath under biaxial stretching has been characterized using the LDH test. The leaf material is found to have high formability with limit strains (~0.4) comparable to that of ductile sheet metals. A strong beneficial effect on formability is demonstrated by hydration treatment of the leaf just prior to the deformation processing. The hydration produces as much as a 500% increase in the forming limit strain compared to that of a dry sheath, while simultaneously lowering the forming force by as much as 85%. Smaller improvements in formability are observed under stepwise loading, low deformation rates and by turning the sheath material "inside-out". The results also show that the Limiting Dome Height test can be a valuable tool for screening the forming response of plant materials.

Besides highlighting the potential for optimizing manufacturing of palm leaf products, the results suggest wide-ranging opportunities for plant leaf materials in manufacturing of eco-friendly foodware products using deformation processing routes.

Acknowledgments

The authors would like to acknowledge B.V. Prashanth Kumar for arranging an industrial visit to Omkara Ventures, Tumkur, India and for showing us the commercial areca-leaf forming process. The work was supported in part by NSF CMMI Award 2100568.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declarations

Conflict of interest

We declare we do not have any conflict of interest.

References

1. R. Geyer, J.R. Jambeck, K.L. Law, Production, use, and fate of all plastics ever made. Sci. Adv. **3**(7), e1700782 (2017)
2. P. Kalita, U.S. Dixit, P. Mahanta, U.K. Saha, A novel energy efficient machine for plate manufacturing from areca palm leaf sheath. J. Sci. Ind. Res. **67**, 807–811 (2008)
3. V. Raghavan, H.K. Baruah, Arecanut: India's popular masticatory—history, chemistry and utilization. Econ. Bot. **12**(4), 315–345 (1958)
4. Staples GW, Bevacqua RF. *Areca catechu* (betel nut palm). In Species Profiles for Pacific Island Agroforestry (Permanent Agriculture Resources (PAR), Holualoa, 2006), pp. 1–17.
5. A.L. Andrady, Microplastics in the marine environment. Mar. Pollut. Bull. **62**(8), 1596–1605 (2011)
6. D.P. Mohanty, A. Udupa, A.R.A. Chandra, K. Viswanathan, J.B. Mann, K.P. Trumble, S. Chandrasekar, Mechanical behavior and high formability of palm leaf materials. Adv. Energy Sustain. Res. **2**(4), 2000080 (2021)
7. L.J. Gibson, M.F. Ashby, B.A. Harley, *Cellular Materials in Nature and Medicine* (Cambridge University Press, Cambridge, 2010)
8. P.B. Tomlinson, *The Structural Biology of Palms* (Oxford University Press, Oxford, 1990)
9. S.P. Keeler, W.A. Backhofen, Plastic instability and fracture in sheet stretched over rigid punches. ASM Trans. **56**(11), 25–48 (1963)
10. Z. Marciniak, J. Duncan, *Mechanics of Sheet Metal Forming* (Edward Arnold, London, 1991)
11. R.C. Hill, A theory of the plastic bulging of a metal diaphragm by lateral pressure. Philos. Mag. **41**(322), 1133–1142 (1950)
12 J.R. Davis (ed.), *Copper and Copper Alloys* (ASM International, Materials Park, 2001)
13 S.L. Semiatin (ed.), *Metalworking: Sheet Forming* (ASM International, Materials Park, 2006)