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Development of wet-laid nonwoven from pineapple leaf fibre for sustainable flushable wipes

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This research aims to explore a sustainable way of utilizing pineapple leaf fibres (PALF) to develop flushable wipes with a higher disintegration percentage. The suitable fibre extraction method has been determined, and the extracted PALF is subjected to steam-explosion using a sodium hydroxide (NaOH) solution with varying NaOH concentration, pressure, and time. Flushable wipes are manufactured using the wet-laid nonwoven technique from steam-exploded pineapple leaf microfibres. From SEM analysis, it is observed that the fibre bundles are separated into individual micro-sized fibres (3–4 μm) after steam explosion. The effect of process parameters in steam explosion treatment on disintegration percentage with respect to shaking speed and time has been explored and then compared with commercial products. The result indicates that the micro-fibres obtained by steam explosion of PALF with 1.5% NaOH at 25 psi for 60 min demonstrate excellent flushability properties (~96.67% disintegration), which exceeds the commercial product by 27.62%. This suggests a potential to meet the EDANA/INDA flushability standards, making it a viable good flushable wipe that won't cause drainage clog. This research concludes that the steam-exploded PALF is found to be beneficial in the hygiene and health care segments for the production of emerging and essential flushable wipes, taking advantage of its unique property of fibrillation on steam explosion.

Keywords: Flushable wipes, Microfibres, Pineapple leaf fibre, Steam explosion, Wet laid nonwoven

1 Introduction

There is a high demand for sustainable, eco-friendly fibres with comparable performance to conventional ones. Natural fibres offer renewable, biodegradable alternatives to synthetic materials, supporting sustainable development goals (SDG 12 and 13). Several researchers have been engaged in exploring the utilization of natural fibres due to their relative affordability, recyclability, and competitive strength per weight of material[1]. According to 2023 UNFAO database, pineapple, a major global fruit, had a world production of 28.6 million metric tons in 2021, with the Philippines, Costa Rica, and Indonesia leading. India, producing 1.8 million tons, is among the top ten cultivators. Vazhakulam in Kerala, known as "pineapple city," is notable for its large-scale pineapple farming[2].

A mature pineapple plant has 40–80 leaves, with 22 leaves per kilogram Leaves (1-1.5 years old) are used to extract pineapple leaf fibre, which is white, lustrous, and ten times coarser than cotton. PALF boasts excellent mechanical and sustainable properties, removing formaldehyde, resisting bacteria, and repelling mites[3]. In the Philippines, PALF is utilized for manufacturing artificial leather and is predominantly used in the field of technical textiles as reinforcing material for composite manufacturing. It reinforces plastic matrices cost-effectively and is used in India for yarns, fabrics, and handicrafts[4,5].

PALF is employed in linings for shorts, mats, rugs, bags, blankets, thermal and acoustic insulating materials[6]. From several studies, it has been found that PALF finds applications in various sectors, including the production of paper[7–10], nanocellulose[11–13], nanocrystals[14], composites[15], geotextile[16], artificial leather[17], acoustic absorbers[18,19], etc. Flushable wipes are highly engineered to be strong at the point of use but rapidly lose strength upon flushing. To be deemed flushable, wipes must clear toilets and drainage as directed, be compatible with wastewater systems without causing blockages, and remain unrecognizable in effluent and treated sludge for soil application[20].

One study had created a wood pulp composite spun-laced nonwoven and established a flushability testing method[21]. Another had constructed a mathematical model for understanding the

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disintegration of flushable solid products, highlighting their unique disintegration characteristics[22]. Another study assessed various toilet papers' disintegration potential in sewer systems via experiments and simulations, estimating disintegration rates and distribution ratios[23]. Additionally, an industry technology roadmap (ITR) for flushable, pre moistened nonwoven wipes had been outlined, addressing market, infrastructure, technology, and regulatory aspects crucial for sector growth and sustainability[24]. Collectively, these studies aimed to enhance understanding, develop effective products, and strategize for industry growth, covering product disintegration, behavior in sewer systems, and industry roadmap creation for sustainable development.

The data above indicate that an excellent natural resource is being unnoticed without its utilization in textiles. Instead of contributing to global warming by burning agricultural waste conventionally, the pineapple leaves can be converted into versatile fibres known as pineapple leaf fibres. However, current extraction methods face a challenge in low fibre yields of less than 3%, making the market potential of PALF unstable in the textile sector. This study focused on finding a suitable fibre extraction methodology for extracting PALF from discarded leaves and also on the development of flushable wipes using pineapple leaf microfibres by wet-laid technology. Utilizing PALF for sustainable product development creates an additional income opportunity for cultivators aligning with SDG 1 of eradicating poverty.

2 Materials and Methods

2.1 Fibre Extraction Method

Around 200 pineapple leaves were utilized for preliminary investigation and then around 2 tons for bulk extraction of fibres from Vadakancherry, Kerala State. Various trials were undertaken to extract fibres from pineapple leaves using a rolling mill, sugarcane juice extractor, and banana fibre extractor. Pineapple leaf processed through the rolling mill extracted out the water as well as the pulpy material within the leaves, but it necessitates additional treatment with alkaline degumming to remove the greenish residue. In another method, spirally grooved rollers resulted in better water extraction but caused more fibre damage. The most successful method was found to be the banana fibre extractor, which efficiently removed pulpy material without damaging the fibres, resulting in a higher yield.

2.2 Bulk Fibre Extraction

The bulk fibre extraction process from various stages of operation includes collection of pineapple leaves from the fields, machine scraping of the leaf, cutting off the hand-held butt end of the leaf from the fibre bunch, drying the fibres, staple cutting, and fibre cleaning.

Machine Scraping of Leaves– Pineapple leaves were fed tip-end into the machine by hand, and fibres are produced through the nibbling action of the raspador (Fig. 1). This process was followed by scraping upon withdrawal of the leaf or fibre in the reverse direction. As a leaf was introduced, the knives progressively smash and chip off the leaf tissue at closely spaced intervals against the feed roller, and only a few centimeters or even millimeters of the leaf being decorticated at any one moment. The crushed portion continued to be beaten for as long as the leaf remained inside the raspador. When the leaf was withdrawn, knives commence to act as scrapers, removing the leaf pulp down the length of the bundle.

The extraction process was carried out after a number of trials, considering variables such as the angle of the leaf inserted inside the extractor, the number of leaves to be inserted inside the extractor for effective removal of pulpy materials from the fibre, the rate of feed of the leaves inside the extractor, and the motor speed was kept constant. After numerous trials, it was decided to feed a bunch of four leaves, one over the other, into the machine at 90° to the scrapping roller, at an average feed rate of 2 cm/s, so that the fibre bundles are freed from greenish pulpy matter, preserving the length of the fibre with minimal fibre loss along with the debris.

Cutting off Hand-held Butt End of Leaf from Fibre bunch– After extracting the fibre in the machine, the hand-held portion of the leaves, ranging in length

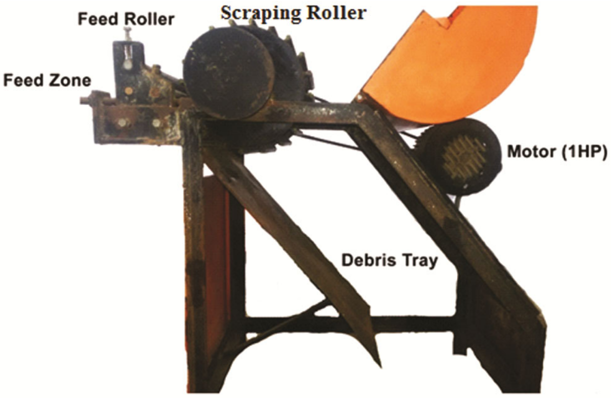

Fig. 1 — Extraction machine based on raspador principle

from 7 cm to 10 cm, was cut and removed manually to obtain the only fibre part of the leaves.

Drying Fibres– The wet fibre strands obtained from the cutting process were dried in the sunlight, followed by shade drying. When the wet fibre was left in the shade for drying after extraction, the development of blackish fungus was observed. To avoid this discoloration, the wet fibre was first dried in the sunlight for 2–3 h and then dried under shade. It was also observed that prolonged exposure of fibre to sunlight resulted in browning of fibre with strength loss.

Staple Cutting– The extracted fibre lengths ranged from 17 cm to 80 cm, were cut into 4-inch staple lengths using the jute staple cutter available at PSG Techs COE Indutech, Neelambur.

Fibre Cleaning– Extracted PALF contained more than 25% of trash particles by weight with most of the particles being well adhered to the fibres. Therefore, the fibres were cleaned using a coir bale opener after manually removing large dried leafy substances.

2.3 Chemical Treatment of Extracted PALF

Degumming Process– The degumming of extracted PALF involved varying concentration of alkali, temperature, and time. The PALF was treated in a bath containing 2, 5, 10, and 18% NaOH (owf) solution with a material-to-liquor ratio of 1:25 at 80 °C for 30 min. Subsequently, the treated sample was rinsed in hot and cold water and dried at 27 °C[25]. Among the four concentrations tested, 5% NaOH yielded optimal degumming results without compromising the fibre's strength ensuring better removal of pulpy material from the fibre's surface. The density of the degummed fibre was found to be 1.52 g/cc, using a density gradient column.

Steam Explosion– Through the literature survey[11,12], it was realized that during the steam explosion of PALF, substantial breakdown of the lignocellulosic structure took place, resulting in improved defibrillation. The raw pineapple leaf fibres were treated with NaOH solution with a material-to-liquor ratio of 1:10 in a high temperature high pressure (HTHP) beaker dyeing machine. During the process, three parameters were varied, viz., NaOH concentration (1.5, 3, and 4.5%), pressure (15, 20, and 25 psi), and time (30, 60, and 90 min) to study the disintegration percentage of wipes after flushing. The fibres were removed from the beaker dyeing machine and rinsed in hot water until they were free from alkali. The rinsed water was drained through a fine mesh to trap the floating fibres. The rinsed fibre mass was gently squeezed, split into small bunches, and air dried.

2.4 Development of Flushable Wipes– Wet Laid Technique

Flushable wipes were prepared using the wet-laying technique from the steam-exploded pineapple leaf microfibres. A prototype wet-lay apparatus was fabricated by utilizing a concept from the literature[26] using polyvinyl chloride (PVC) pipe fittings and plywood, as shown in Fig 2(a).

Fibre Dispersion– One gram of the dried steam-exploded fibres was evenly spread as homogeneously as possible in two liters of cold water (at a rate of 0.5 g/L)[27,28], as shown in Fig. 3.

Principle of Operation– With the valve closed, the water column was filled up to the level of the drainage mesh. The chamber, above the drainage mesh, was filled with the diluted fibre dispersion [Fig. 2(c)]. The valve was then opened, and the water in the column rushed out, drawing behind the water in the fibre dispersion. The fibres were deposited on the drainage mesh (mesh size 2 mm in diameter) as the water drained out. The rapid drainage of water created a stack of randomly oriented microfibres[29].

False Drainage Mesh Arrangement– In fact, the wet laying of fibres was carried out on a false drainage mesh arrangement composed of nylon bolting cloth backed with a needle-punched polyester nonwoven. It was fastened intact to one end of a circular PVC pipe, which was then inserted into the chamber above the drainage mesh and placed over it later. The diluted fibre dispersion was poured into the PVC pipe over the false drainage mesh. This arrangement facilitated the easy removal of the wet-laid sample without disturbing the apparatus.

Dewatering– The removal of excess water from the wet-laid nonwoven was accomplished by placing absorbent tissue papers over the nonwoven sheet and gently rolling a stainless-steel roller over the tissue paper to facilitate water imbibitions. Then, the sheets were air dried.

2.5 Characterization of Developed Nonwoven

2.5.1 SEM Analysis

The morphology and surface topography of raw, degummed, and steam-exploded fibres were examined using scanning electron microscopy (SEM) at an emission current of 58 microamperes and an acceleration voltage of 5 kilovolts.

2.5.2 Flushability Testing

The test method was first introduced by INDA and EDANA, and there were only two labs in the world to

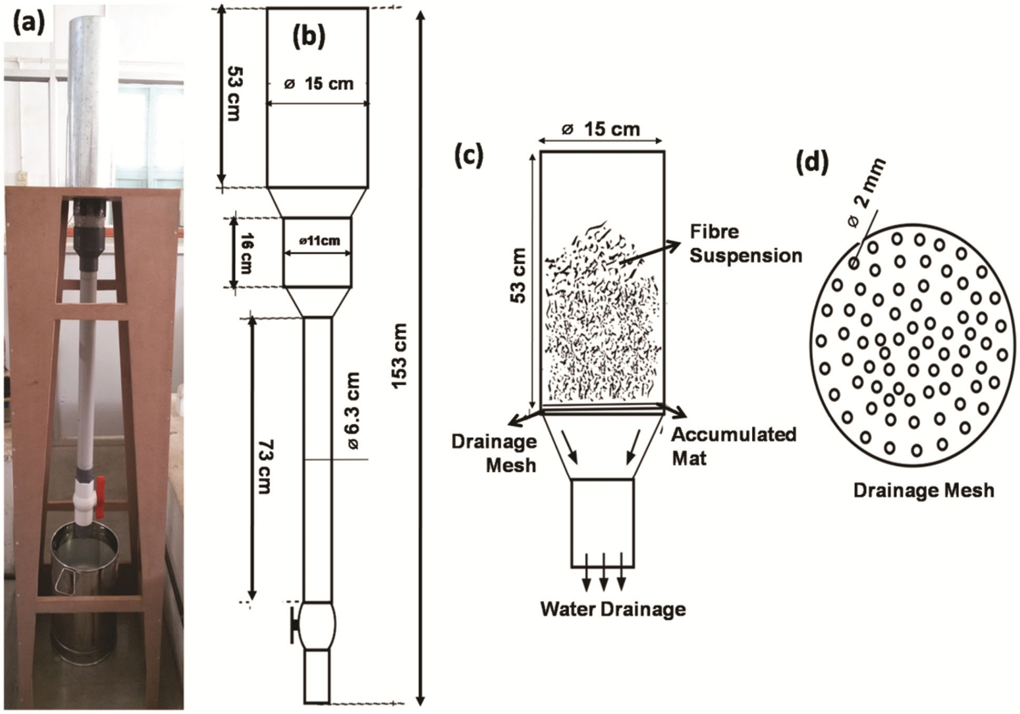

Fig. 2 — Wet-laying technique (a) apparatus set up, (b) schematic diagram, (c) fibre dispersion and (d) cross-sectional view of drainage mesh

Fig. 3 — Stages of fibre dispersion

perform the tests. The orbital shaker table was used to test the dispersibility of the wipes produced, following closely the procedure outlined in the previous study[21].

Square sample (2 cm × 2 cm) was weighed as $W_1$and then placed in 250 mL conical flasks that were filled with 100 mL of tap water. The flasks were then placed on a sub zero orbital shaker table and stirred. The samples were removed at the scheduled time and run through an 8 mm sieve at a distance of 15 cm in 8–10 s. After sieving and air drying, the remaining materials were placed into PET containers and oven dried. W_2 weights were assigned to the dried samples. Following equation was used to calculate the disintegration percentage, which was used to determine the outcome:

$$\text{Disintegration (\%)} = \frac{W_1 - W_2}{W_1} \times 100 \qquad \ldots(1)$$

In this study, parallel tests were conducted at different shaking speeds and different shaking time. Additionally, a commercial flushable wet wipe sample, produced by one of the leading corporations, was purchased from the market and tested under the same afore mentioned conditions.

3 Results and Discussion

3.1 SEM Analysis

Surface morphology of raw, degummed and steam-exploded fibres has been studied using SEM, as illustrated in Fig. 4. The raw fibre surface exhibits slight roughness due to the presence of an outer noncellulosic layer comprising cementing materials, such as lignin, hemicelluloses, pectin, wax and oil, with fibres being adhered closely [Fig. 4(a)]. After degumming, fibre surfaces become smoother with the removal of certain impurities [Fig. 4(b)].

After steam explosion, the fibre bundles are separated into individual micro-sized fibres of 3-4 µm, as shown in Fig. 4(c). The steam-exploded PALF fibres exhibit weight loss ranging from 40% to 50%, depending on the process parameters, attributed to the dissolving out of the hemicellulose and lignin components present in the raw fibre[11]. The removal of hemicelluloses and lignin with some other extracts during the alkali treatment is the primary reason for this observation.

3.2 Wet Lay Process

The steam-exploded fibres, when soaked and agitated in the cold tap water of required quantity, disperse homogeneously to a satisfactory level. After it is settled, the dispersion of fibres remains more or less uniform with little below the surface of the water. This may be attributed to the formation of hydrogen bonding between water molecules and the fibre, as steam explosion results in a higher availability of hydroxyl groups distributes both on the surface of the cellulose crystals and in the non-ordered regions[11]. Additionally, the property of cellulose acquiring a negative charge when immersed in water contributes to the dispersion of the short fibres of high cellulose content by mutually repelling each other.

After the wet laying process, the moist and dried nonwoven sheets exhibited sufficient strength to be used as moist or dry wipes. This favorable property may be attributed to the increase in strong inter-fibrillar attraction via hydrogen bonding among the surface hydroxyl groups of cellulose during the drying process[12] and also due to the varying length of microfibres. Since, in pure cellulose, each unit has three free hydroxyl groups, the moisture absorption rate is higher. The areal density of the dry nonwoven sheet is 60-65 gsm, which is comparable to the existing commercial products (60-70 gsm).

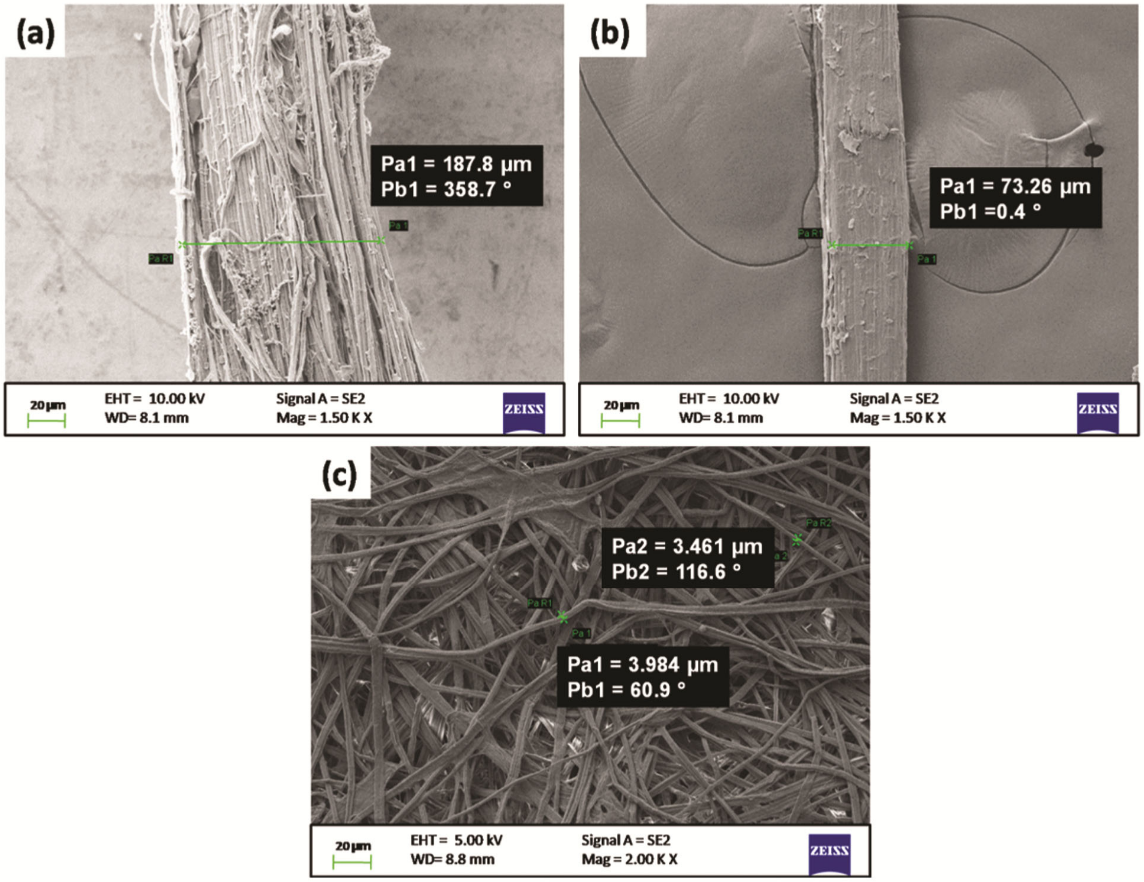

Fig. 4 — SEM analysis of (a) untreated, (b) degummed and (c) steam-exploded PALF

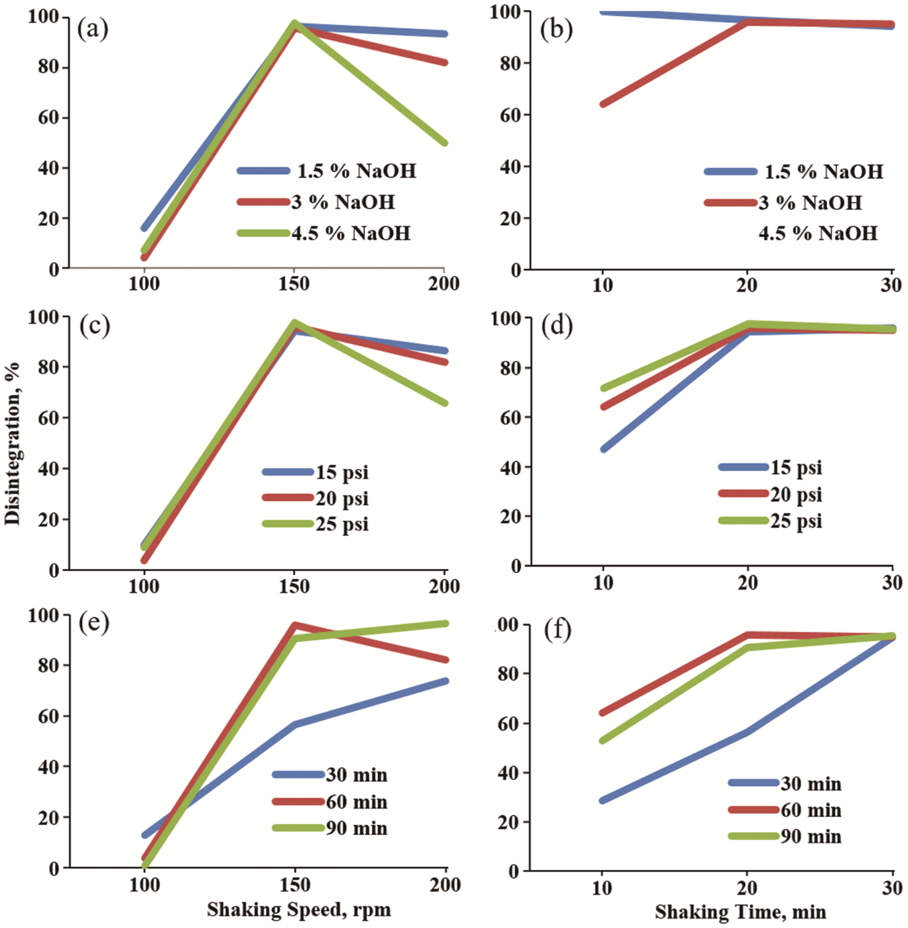

Fig. 5 — Effect of (a - b) NaOH concentration, (c-d) pressure and (e-f) explosion time on disintegration percentage, with respect to shaking speed and shaking time, respectively

3.3 Effect of NaOH % in Steam Explosion on Flushability Testing

Varying Shaking Speed– The effect of NaOH concentration (1.5, 3 and 4.5%) in steam explosion on flushability has been investigated for 20 min at three different shaking speeds (100, 150 and 200 rpm). Figure 5 (a) shows that the disintegration percentage of the nonwoven rises along with the increase of speed and then decreases slightly, which can be attributed to the fibrillation and intertwining of fibres. At lower shaking speeds, gentle agitation allows the highly fibrillated fibres to disperse more easily. However, as the speed increases, the agitation becomes more vigorous, causing the shorter fibrillated fibres to intertwine or "rope" together due to the swirling action. With higher NaOH concentrations during steam explosion, the fibres become more finely fibrillated, which, when subjected to very high shaking speeds, leads to increased intertwining or roping of these fine fibres. This phenomenon impedes the disintegration process as the entangled fibres resist separation when subjected to excessive agitation. Therefore, while moderate shaking aids dispersion, very high speeds result in fibre entanglement, reducing the disintegration percentage.

Varying Shaking Time– The effect of NaOH concentration in steam explosion on flushability has been investigated under the time of 10, 20 and 30 min at constant speed of 150rpm. Figure 5(b) clearly shows that the disintegration percentage increases when shaking time is increased. The increase of shaking time from 20 min to 30 min does not show increased disintegration; instead it slightly decreases irrespective of the varying alkali concentration employed in steam explosion. The higher disintegration percentage under all the times of the sample steamed with 1.5% NaOH may have been attributed to the less inter-fibrillar attraction. This shows an optimal shaking duration beyond which further time might not significantly improve the disintegration of NaOH-treated samples.

3.4 Effect of Pressure in Steam Explosion on Flushability Testing

Varying Shaking Speed– The investigation on pressure variations (15, 20, and 25 psi) during steam explosion and its impact on flushability, examined under different shaking speeds, reveal a similar trend. Figure 5(c) illustrates an increase in the nonwovens' disintegration percentage with increasing speed, followed by a moderate decrease. The increased pressure during steam explosion increased fibrillation into microfibres. However, these microfibres, with an increase in free hydroxyl groups on their surfaces due to higher pressure, appear to rope or intertwine in the swirling water instead of disintegrating. This phenomenon suggests that while increased pressure promotes fibrillation, excessively high pressure might have led to greater roping of microfibres, hindering their effective disintegration in water.

Varying Shaking Time– The study of pressure variation keeping shaking speed constant and its impact on flushability has been investigated under the time duration of 10, 20 and 30 min. Figure 5(d) clearly shows that the disintegration percentage increases when shaking time is increased. Elevated pressure during steam explosion potentially promotes greater fibre breakdown into smaller fragments, aiding their disintegration during shorter shaking times. However, beyond a certain threshold, additional pressure might not have significantly improved disintegration within the shorter shaking durations, explaining the plateau observed at higher shaking times and pressure levels.

3.5 Effect of Time in Steam Explosion on Flushability Testing

Varying Shaking Speed– The effect of time variation in steam explosion and shaking speed on flushability has been investigated under the speed of 100, 150 and 200 rpm for 20 min. Figure 5(e) shows that the disintegration percentage of the nonwoven rises along with an increase of speed of shaking and an increase in steam explosion time. Samples stirred at higher speed were dispersed more easily than those stirred at slower speed. This increase aligns with the longer steam explosion time, suggesting that extended steam explosion times possibly contributes to enhanced microfibre fibrillation without excessive dissolution of cementing components like hemicellulose and lignin. Consequently, higher shaking speeds may have offered sufficient agitation to facilitate fibre disintegration.

Varying Shaking Time– The effect of time variation in steam explosion and shaking time on flushability is investigated under the time of 10, 20 and 30 min at 150 rpm. Figure 5(f) reveals that beyond 20 min of shaking, further duration doesn't significantly augment disintegration. Interestingly, at shorter shaking times (10 min), the disintegration percentage rises with increased steam explosion time, indicating that longer steam explosion durations cause disintegration. Conversely, the fibres subjected to shorter steam explosion times may have possessed a stickier nature, requiring extended shaking times for effective disintegration. This trend suggests an optimal balance between steam explosion and shaking times for achieving enhanced fibre disintegration without excessive dissolution or stickiness.

3.6 Comparison of Developed Nonwoven with Commercial Product

Figures 6(a) and (b) reveal a consistent disintegration percentage of the commercial product across varying shaking speed and time. It is observed that though the disintegration percentage is comparatively good, one of the two plies in the product remains intact without disintegration in all the three-shaking speeds and times.

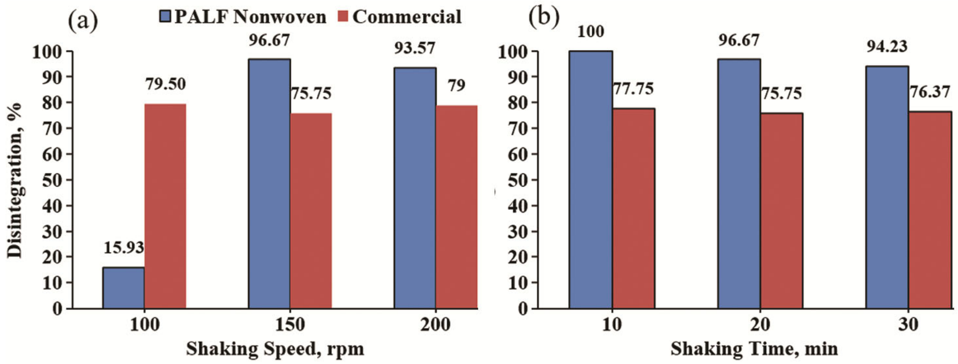

Fig. 6 — Comparison of developed nonwoven with commercial product on disintegration percentage with respect to (a) shaking speed and (b) shaking time

Comparatively, the developed sample exhibits promising efficiency, boasting a disintegration percentage of 96.67% which is 27.61% higher than the commercial sample at 150 rpm for 20 min. This substantial increase in disintegration performance suggests that the developed sample could be a significantly superior alternative to the commercial product. However, considering the consistent intact ply in the commercial product, further investigation and refinement are essential to ensure complete and uniform disintegration across all layers of the developed sample for its optimal use as a flushable product.

4 Conclusion

Fibres from fresh pineapple leaves can be economically extracted using the available banana fibre extractor. Immediate cleaning of the extracted fibre helps to reduce adhering impurities. Degumming of the fibres improves handling and aesthetic properties. Microfibres for flushable wipes were obtained by steam explosion of PALF with a 1.5% NaOH solution (fibre to liquor ratio 1:10) at 25 psi for 60 min. The wipes made from these fibres disintegrated around 95% at the shaking speed of 150 rpm in 20 min, ensuring they will not clog drainage pipes. Moreover, wipes made from these fibres may exceed the EDANA/INDA flushability standards. The shaking speed and the shaking time are critical controlling factors for testing the flushability. As one of the two plies of the market product remains intact without disintegration, there is a possibility of clogging of wastewater conveyance upon flushing the product into the toilet, meeting the norms of disintegration percentage.

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