

**PINEAPPLE RESIDUES: A TRANSITION FROM REMNANTS TO THE
REALM OF A BASKET OF CHEMICALS AND VALUE-ADDED
PRODUCTS**

A PROJECT REPORT
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THE AWARD OF THE DEGREE
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IN
CHEMISTRY

Submitted by

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CANDIDATE'S DECLARATION

We, Eshita Pasreja (2k21/MSCCHE/18) and Dishika Garg (2k21/MSCCHE/14) of M.Sc. Chemistry, hereby declare that the project titled “Pineapple residues: a transition from remnants to the realm of a basket of chemicals and value-added products” which is submitted by us to the Department of Applied Chemistry, Delhi Technological University, Delhi in partial fulfilment of the requirement for the award of the degree of Master of Science, Chemistry is original and not copied from any source without proper citation. This work has not previously formed the basis for the award of any Degree, Diploma Associateship, Fellowship or other similar title or recognition.

Place: Delhi

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CERTIFICATE

I hereby certify that the Project titled “Pineapple residues: a transition from remnants to the realm of a basket of chemicals and value-added products” which is submitted by Eshita Pasreja, 2k21/MSCCHE/18 and Dishika Garg, 2k21/MSCCHE/14 of Department of Applied Chemistry, Delhi Technological University, Delhi in partial fulfilment of the requirement for the award of the degree of Master of Science, Chemistry, is a record of the project work carried out by the students under my supervision. To the best of my knowledge this work has not been submitted in part or full for any Degree or Diploma to this University or elsewhere.

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ABSTRACT

The major part of food waste in our surroundings is produced due to the processing of fruits and vegetables. To recycle this waste, a considerable amount of money is required and as a result these pollutants are either dumped in open spaces or else burned which tends to intensify the pollution problems. This project report provides the information regarding the potential of pineapple waste including the left-over peels, core, stem and crown portions to generate value-added compounds and the techniques that can be used to extract these compounds from the by-products have also been assembled from literature. Further, the applications of these compounds in numerous fields were extensively explored. The highly nutritious profile of pineapple waste including its high carbohydrate content makes it a suitable substrate for manufacture of important chemicals such as organic acids, essential oils, nanocellulose, xylooligosaccharides, polyphenols, bromelain and others that may find use in the food industry, production of aerogels, fibers, biofuels, animal feed, production of nanoparticles, heavy metal intoxication, bioadsorbent and a variety of clinical application. Such utilisation of waste would be immensely helpful in the reduction of environmental issues and would also provide an innovative way to manufacture useful compounds out of waste.

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ESHITA PASREJA

DISHIKA GARG

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CHAPTER - 1

INTRODUCTION

1.1 General aspects of *Ananas cosmosus*

The waste generated from fruits and vegetables which is also known as agricultural wastes (or agri-waste) are the source of numerous chemicals. The lack of proper harvesting method, logistical management, storage facilities and improper transportation results in the production of huge amount of agri-waste (Roda and Lambri, 2019). Out of the many types of agri-waste, the waste generated from fruits and fruit processing industries constitutes noticeable amount.

The tropical fruit *Ananas comosus*, commonly referred as pineapple, belongs to the Bromeliaceae family. The pineapple plant is herbaceous, monocotyledonous and constitutes the stem, leaves, peduncle, crown, shoots and adventitious roots as its basic morphological components (Hossain, 2016). The initial leaf sprout is tender and the leaves gradually become stiffer and acquires a sword-shaped structure that wraps around the fruit (Fouda Mbanga and Tywabi Ngeva, 2022). Acidic loams, sandy loams and clay loams all support the growth of high-quality pineapples in warm, humid climates with sunny days and chilly nights, with the ideal soil pH range between 4.5 and 6.5 (Hossain, 2016). According to the data from Food and Agriculture Organization (FAO), 28 million tonnes of pineapple have been produced globally in 2020 and the Philippines, Costa Rica, Brazil, Indonesia, and mainland China make up the top five nations that produce pineapples (Figure 1.1) ("FAOSTAT," 2020). The five groups of commercial pineapple cultivars are Cayenne, Queen, Spanish, Pernambuco, and Mordilona. Despite considerable variability among the types within each class, the diverse pineapple cultivars are divided into four major classes for international trade: "Smooth Cayenne", "Red Spanish", "Queen" and "Abacaxi". Smooth Cayenne has a distinctively smooth exterior and is the most grown cultivar for fresh fruit and canning. Abacaxi has distinctive fruits that are long, white, translucent and delicious. Although it is the most exquisite type of pineapple, due to its fragility, it cannot be canned. Queen has a dwarf plant with short, extremely spiky, dark purplish-green leaves that have high sugar content. Its fruit is conical, deep yellow and has deep eyes; a thick cut is needed to entirely remove the peel; when mature, it has a wonderful fragrance and flavor. Red Spanish fruit has deep eyes and is often rounded with an orange-red exterior. When fully grown, the fruit is robust and readily and

neatly snaps off at the base during harvest. In addition to these, it is known that there are numerous hybrid pineapple cultivars (Banerjee et al., 2018; ITFNet, 2016).

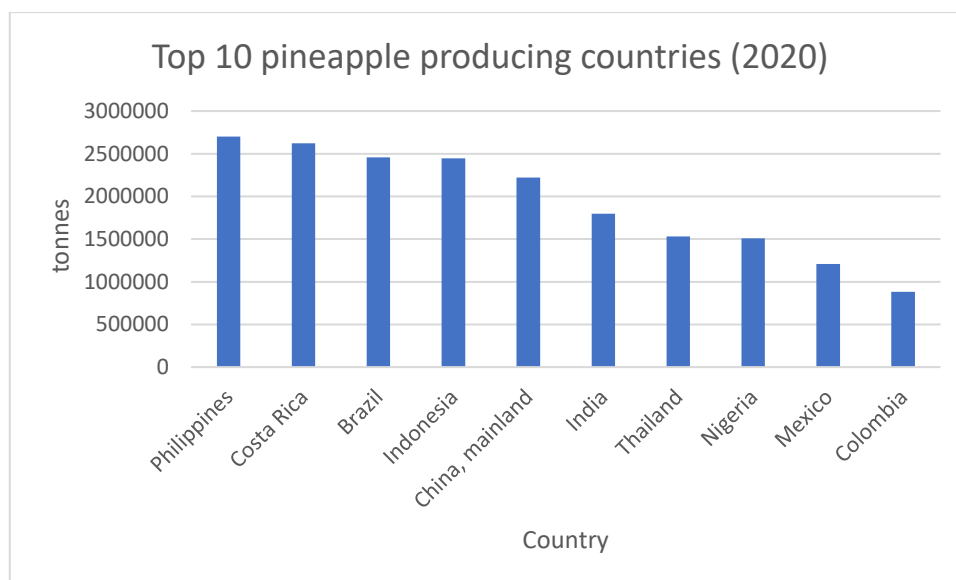


Figure 2.1: Leading countries in pineapple production (“FAOSTAT,” 2020)

1.2 WASTE GENERATION FROM PINEAPPLES

Waste from pineapples have been categorized as pineapple processing waste (PPW) and pineapple on-farm waste (POFW). After pineapples have been harvested, POWF contains the leftover leaves, stems and roots in the fields. Then the pineapples are processed which includes slicing, pulping and juicing (Banerjee et al., 2018). Waste produced by the industries that process pineapples includes leftover skin, peel, pulps, stems and leaves (Rabiu et al., 2018). The removal of the crown is the initial processing stage, which is followed by the simultaneous removal of the peels and core and the waste produced at this stage is PPW (Banerjee et al., 2018). The main reason pineapples are grown is for their fruit, which is eaten either fresh or in canned fruit and juice form. Besides this, huge quantities of by-products are produced during the processing of pineapple, with peels accounting for 29–42% (w/w), core accounting for 9–20% (w/w), crown accounting for 2.7–5.9% (w/w) and stem accounting for 2.4–6.8% (w/w). All these by-products contain cellulose, hemicellulose and lignin (Figure 1.2) (Rico et al., 2020; Sukruansuwan and Napathorn, 2018). There is significant interest in leftover pineapple peels, crown and other by-products to convert it into value-added products. By-products such as peels, leaves, seeds and leftover flesh

account for up to 50% by weight of the overall pineapple crop. The primary components of pineapple waste include the remaining pulp, the peels, the stems and the leaves. During industrial processing, pineapple fruits produced 55% of leftovers, which included peel (40%), bagasse (23%) and stem and crown portions (14%) (Rajapaksha et al., 2019). The increase in the amount of processed pineapple leads to enormous waste generation (Upadhyay et al., 2010). Unrefined pineapple industry effluent contains higher concentrations of carbohydrates like sucrose, glucose and fructose, which may lead to environment pollution when released into the rivers. The pineapple industry's wastewater discharge results in high level of suspended solids (SS), chemical oxygen demand (COD) and biochemical oxygen demand (BOD). High COD concentrations in wastewater is harmful to biological life and have an impact on the aquatic environment. Nearly every step of the production process in the pineapple refining industry requires a significant amount of water as due to which a lot of solid and liquid waste is produced which accounts for serious environmental issues (Fouda Mbanga and Tywabi Ngeva, 2022).

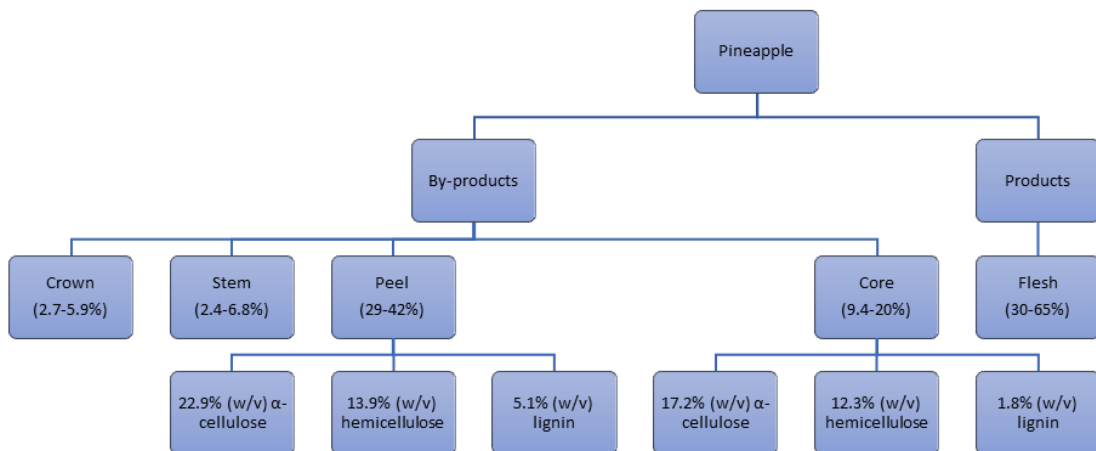


Figure 1.2: Fruit processing (Rico et al., 2020; Sukruansuwan and Napathorn, 2018)

A number of articles have been published on utilisation of pineapple waste (Figure 1.3) (“Dimensions,” 2022).

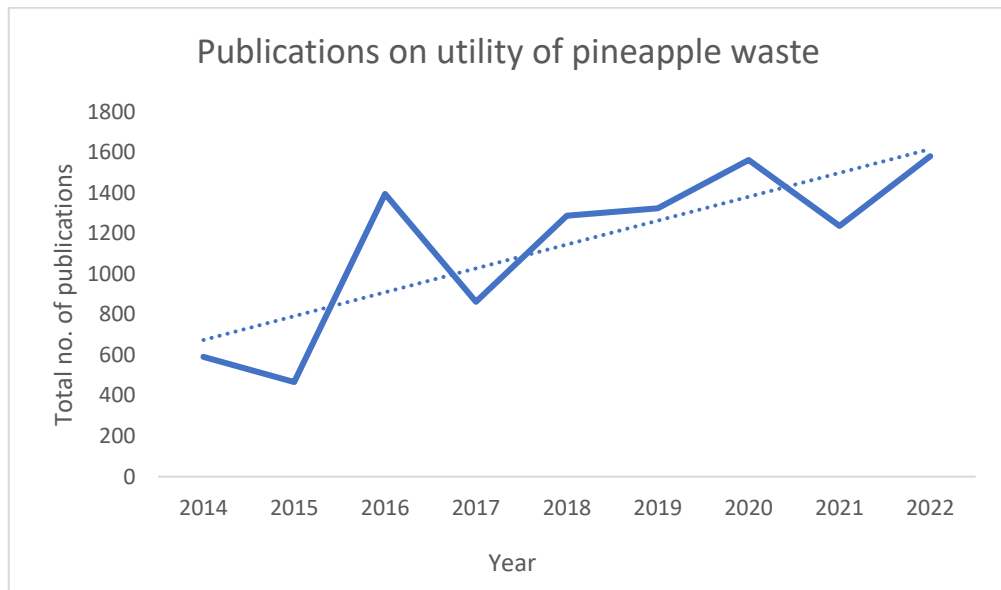


Figure 1.3: Publications on utility of pineapple waste (“Dimensions,” 2022)

CHAPTER - 2

PINEAPPLE WASTE

UTILISATION

2.1 MOLECULES EXTRACTED FROM PINEAPPLE WASTE

The pineapple waste is a valuable substrate with significant potential if appropriate processes and technologies are applied to transform its various components into value-added products (Roda and Lambri, 2019). Due to high sugar content, antioxidants, bioactive compounds and bromelain enzyme, pineapple by-products have a significant potential for use in the production of organic acids, polyphenols, bromelain, adsorbent as well as biofuels and biogas (Aili Hamzah et al., 2021). Pineapple pulp has the greatest values in terms of protein, ash, and fibre content (Pardo et al., 2014). Pineapple waste served as both, a carbon source for acid fermentation and a beneficial nutrient for bacterial growth (Abdullah and Mat, 2008). The PW's non-reducing sugars, carbohydrates and protein are used as a nutritional medium for the growth of microorganisms and yeast fermentation, which can further produce ethanol and single cell protein (Hemalatha and Anbuselvi, 2013). The prebiotic effects of pineapple peel flour can promote the development of probiotic bacteria in the gut. Vinegar and vanillin were produced from pineapple waste (Cheok et al., 2018; Roda et al., 2017).

2.1.1 *Organic acids*

Pineapple waste can be utilized to produce organic acids namely citric acid, succinic acid, lactic acid, acetic acid, and oxalic acid by the process of fermentation employing different microorganisms for its production (Figure 2.1, Table 2.1).

Citric acid

Citric acid, having molecular formula $C_6H_8O_7$ is a weak organic acid that contains three carboxyl ($R-COOH$) groups (Berovic and Legisa, 2007). The citric acid is produced by solid state fermentation (SSF) of glucose or sucrose. The process of microbial fermentation is more cost-effective than the chemical methods used for its production. Solid pineapple waste has been utilized as a substrate for the production of citric acid using *Yarrowia lipolytica* through SSF (Imandi et al., 2008). *Aspergillus niger* was also used to produce citric acid from agri-waste and used less expensive raw materials, high product yield and lack of unfavourable reactions (Majumder et al., 2010; El-Holi and Al Delaimy, 2003).

Succinic acid

Succinic acid, 1,4-dicarboxylic acid, having molecular formula $C_4H_6O_4$ and the IUPAC name butanedioic acid is one of the potential compounds that are produced from pineapple waste (Jusoh et al., 2014). High levels of sugar and other macro- and micronutrients are present in liquid pineapple waste, which have been employed as a substrate for the production of succinic acid (Jusoh et al., 2014). Solid pineapple waste, notably the peel portion, was used as a carbon source and fermentation with *Actinobacillus succinogenes* resulted in bio-based succinic acid production (Pathanibul and Hongkulsup, 2021). The liquid pineapple waste was fermented with *Escherichia coli* under aerobic conditions to produce succinic acid (Jusoh et al., 2014). *Aspergillus niger* and *Rhizopus oryzae* produced fermentable sugars by biological hydrolysis of pineapple waste. Then, *Actinobacillus succinogenes* was used to carry out the biotransformation of the solid waste hydrolysate to produce succinic acid (Dessie et al., 2018).

Lactic acid

Lactic acid, hydroxycarboxylic acid having chemical formula $CH_3CH(OH)COOH$ and the IUPAC name 2-hydroxypropionic acid can be produced from pineapple syrup, a by-product of the pineapple processing industry. Pineapple syrup was employed as an inexpensive substrate and through fermentation process using *Lactobacillus lactis* and the enzyme invertase, lactic acid has been produced (Ueno et al., 2003). Pineapple waste streams were utilised as a production medium for lactic acid using microorganisms, *Rhizopus arrhizus* and *R.oryzae* and fungal biomass by single stage simultaneous saccharification and fermentation process (Jin et al., 2005). Liquid pineapple waste was employed as the substrate which was then subjected to a 72-hour fermentation to convert it into lactic acid using *Lactobacillus delbrueckii*. Calcium alginate was employed as the immobilisation matrix by varying temperature and pH conditions (Idris and Suzana, 2006).

Acetic acid

Acetic acid (ethanoic acid) is a carboxylic acid having the molecular formula CH_3COOH . Pineapple waste contains high sugar content which was exploited to

produce acetic acid. PW was saccharified using physical and enzymatic processes and the resulting substrate was fermented with *Saccharomyces cerevisiae* for 7–10 days at 25°C in aerobic conditions. *Acetobacter aceti* was employed as the inoculum and the alcoholic medium was used as a seed broth for the fermentation process and it took roughly 30 days at 32°C to produce acetic acid (Roda et al., 2017). In particular, the fermentation of vinegar involves two steps: first, anaerobic fermentation of fermentable carbohydrates into ethanol by yeasts typically of the *Saccharomyces* genus and then aerobic oxidation of ethanol to acetic acid by bacteria generally of *Acetobacter* genus. High rates of aeration during the process can result in better yield of acetic acid (Umaru et al., 2015).

Table 2.1: Organic acids from *Ananas cosmosus*

Organic Acid	Method of Production	Microorganism	Reference
Citric acid	Fermentation	<i>Yarrowia lipolytica</i> <i>Aspergillus niger</i>	Ayeni et al., 2019; Papagianni, 2007
Succinic acid	Fermentation	<i>Actinobacillus succinogenes</i> <i>Escherichia coli</i>	Jusoh et al., 2014; Pathanibul and Hongkulsup, 2021; Dessie et al., 2018
Lactic acid	Fermentation	<i>Lactobacillus lactis</i> <i>Lactobacillus delbrueckii</i> <i>Rhizopus arrhizus</i> <i>R. oryzae</i>	Datta and Tsai, 1997; Idris and Suzana, 2006; Jin et al., 2005; Ueno et al., 2003
Acetic acid	Fermentation	<i>Saccharomyces cerevisiae</i> <i>Acetobacter aceti</i>	Umaru, 2015; Roda et al., 2017
Oxalic acid	Fermentation	<i>Aspergillus niger</i>	Amenaghawon and Kazeem, 2020

Oxalic acid

Oxalic acid (OA), also known as ethanedioic acid is the simplest aliphatic dicarboxylic acid having the chemical formula $C_2H_2O_4$ (Amenaghawon and Kazeem, 2020). Pineapple juice serves as a carbon source and fermenting it with a filamentous

fungus *Aspergillus niger*, oxalic acid has been produced (Amenaghawon and Kazeem, 2020).

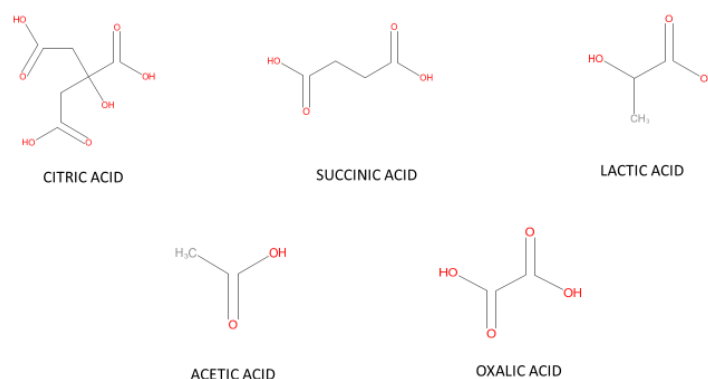


Figure 2.1: Structure of organic acids derived from pineapple waste

2.1.2 Essential oils

Essential oils have been extracted from the peel and fibre portions of pineapple waste. Of the 35 compounds found in the oil, esters (37%), alcohols (29%), aldehydes (9%), ketones (9%), and acids (6%) were the most prevalent compounds in pineapple waste (Barretto et al., 2013). Essential oils were extracted from pineapple waste using three distinct techniques namely hydro-distillation (HD), hydro-distillation with enzyme-assisted pre-treatment (HDEA) and supercritical fluid extraction (SFE) (Mohamad et al., 2019).

Hydro-distillation (HD)

Essential oils from pineapple waste were extracted using hydro-distillation process where pineapple peels and distilled water were added to a round bottom flask connected to the cleverger-type apparatus and once the process was completed, essential oils were collected in the trap (Mohamad et al., 2019).

Hydro-distillation with enzyme-assisted pre-treatment (HDEA):

Hydro-distillation with enzyme-assisted pre-treatment method can be used for the extraction of essential oils from dried pineapple peels by mixing the peels with

acetate buffer followed by addition of acromonium cellulase solution at different enzyme loadings prior to hydro-distillation procedure (Mohamad et al., 2019).

Supercritical fluid extraction (SFE)

SFE was used for the extraction of essential oils from the pineapple peels. The ground-dried pineapple peels were placed in the SC-CO₂ extraction apparatus (Kamali et al., 2015). Then, the compressed CO₂ was pumped into the extractor at the pressure of 180 bar and a temperature of 50 °C and a static period was used to allow contact between the sample and CO₂ solvent followed by dynamic extraction (Kamali et al., 2015). After the extraction, the extracted oil, wax and solid residue were collected, with the release of CO₂ into the air (Mohamad et al., 2019). Distillation using SFE gave the highest oil (0.17%) as compared to distillation using HD and HDEA methods which produced hydrosol instead of essential oils (Barretto et al., 2013). Propanoic acid ethyl ester (40.25%), lactic acid ethyl ester (19.35%), 2-heptanol (15.02%), propanol (8.18%), 3-hexanone (2.60%), and butanoic acid ethyl ester (1.58%) were the main components of the essential oil produced from pineapple peels by the SFE process (Barretto et al., 2013).

2.1.3 Carbohydrates

Xylan-rich lignocellulosic waste from pineapple was used for the production of xylooligosaccharides (Banerjee et al., 2018). Pineapple peel waste was used as a substrate to produce hemicellulose using hydrothermal-assisted alkali extraction technique. The peels were incubated at various alkali concentrations with temperatures varying from 35 °C to a maximum of 65 °C for a time period of 16 hours to extract the hemicellulose. Then, the extracted hemicellulose was enzymatically hydrolysed using endo-1, 4-β-xylanase M1 to produce XOS (Banerjee et al., 2019). Fermentable sugars were produced from sugarcane baggase by enzymatic hydrolysis using a mixture of Cellic CTec2 and Cellic HTec2 and using autohydrolysis technique for pre-treatment followed by mechanical refining technique (Batalha et al., 2015). The fermentable sugars were produced by using pineapple leaf waste that are rich in polymers such as cellulose and hemicellulose. Reducing sugars were produced through enzymatic

means through delignification of pineapple leaf waste using laccase and saccharification employing cellulase-xylanase concoction (Banerjee et al., 2017).

Pectin

Pectin was extracted from pineapple waste using conventional and distinctive methodology (Marić et al., 2018). The ultrasound-assisted extraction (UAE) technique was used to extract pectin from pineapple peel (PP) waste (Shivamathi et al., 2022). Pectin was also extracted using acid extraction and ethanol precipitation (Sarangi et al., 2020). Dried pineapple waste was taken in a beaker and distilled water and sulphuric acid was added to it. The mixture was then stirred continuously with simultaneous heating. It was followed by the filtration process; ethanol was added for the coagulation of filtrate. Pectin was obtained after filtration and was washed with varying concentrations of ethanol (Sarangi et al., 2020). Pectin was also extracted using microwave heating (ME) (Zakaria et al., 2021). The solvent used was sulphuric acid with concentration, 0.5 N H₂SO₄, pH 1.83 and 1:30 (w/v) solid-to-solvent ratio.

Starch

Starch have been extracted from the remnants of pineapple, particularly stem by mechanical extraction using water. Pineapple stem was taken and grounded with distilled water with a weight ratio of 1:1 using a grinder and a paste with crude fibrous material was obtained. Fibrous materials were then filtered out with a cloth. Then, the starch was separated from the extracted compounds using the process of centrifugation. The process was then repeated multiple times until the liquid fraction became clear and was dried in oven and sieved through a mesh screen to obtain starch (Nakthong et al., 2017).

Glucose

Pineapple waste rich in cellulose has been utilized to produce glucose by the process of enzymatic hydrolysis utilising cellulase from *Trichoderma reesei*. The enzyme substrate was incubated at pH 5.5, 50 °C, 120 rpm and 72 h with 10% (w/v) solid loadings of substrate for the production of glucose. The enzyme was inactivated by heating it at 95 °C, followed by centrifugation for the enzyme to settle down and

the supernatant was filtered to determine the glucose concentration using HPLC (Banerjee et al., 2022).

2.1.4 Polyphenols

Pineapple skin contains significant amount of phenolic chemicals (Yahya et al., 2019). Salicylic acid, caffeic acid, myricetin, syringic acid, sinapic acid, ferulic acid etc. were some of the phenolics identified in pineapple (Figure 2.2). Their extraction methods included soxhlet extraction (Madhumeena et al., 2021; Salve and Ray, 2020), supercritical fluid extraction (Salve and Ray, 2020), pressurised solvent extraction (Kaufmann and Christen, 2002), ultrasound assisted extraction (Kumar et al., 2021; Salve and Ray, 2020), microwave assisted extraction (Alias and Abbas, 2017; Salve and Ray, 2020; Vargas-Serna et al., 2022) and autohydrolysis (Batalha et al., 2015; Sepúlveda et al., 2018) (Table 2.1).

Soxhlet extraction

The sample containing pineapple waste was put in the thimble, which was then placed in a distillation flask filled with solvent (Salve and Ray, 2020). Methanol, ethyl acetate, gasoline and their aqueous solvents were found to be the most commonly used solvents for the production of polyphenols from plant sources. Aqueous methanol was identified as a most popular choice due to its high boiling point and its economic availability (Madhumeena et al., 2021).

Supercritical fluid extraction

The polyphenols present in pineapple waste can be extracted using SFE technique. The CO₂ is regarded as an optimal solvent for SFE as its critical temperature is close to room temperature and it has low critical pressure. Dichloromethane (CH₂Cl₂) can be employed as a chemical modifier and aid in the extraction procedure of polyphenols (Salve and Ray, 2020).

Pressurised solvent extraction

The solid or semi-solid sample of pineapple waste is placed in the extraction cell and is filled with the solvent and heated in an oven. It uses diethyl ether and other

organic solvents at high pressures and temperatures that helps to improve the effectiveness of the extraction process. Pressure in the extraction cell is produced by solvent expansion during the heating process (Kaufmann and Christen, 2002).

Ultrasound assisted extraction

The process of ultrasound assisted extraction (UAE) is used to extract polyphenols from pineapple peel (Salve and Ray, 2020). In the cellular structure of the pineapple plant, collapsing cavitation bubble and the sound waves may cause one or a combination of factors that includes localised erosion, pore formation, enhanced absorption, shear force, fragmentation, and swelling index. Shockwaves are produced by collapsing cavitation bubbles and the fragmentation of cellular structure is brought on by accelerated inter-particle collision. The bioactive components become solubilized in the solvent as a result of the rapid fragmentation. Ultrasound causes damage to the plant known as erosion, which is the explosion of cavitation bubble on the surface of the plant. The release of polyphenols is caused by the development of pores during cavitation, a process known as sonoporation. Additionally, the formation and disintegration of cavitation bubbles cause turbulence and shear force, which causes the breaking of cell walls and aids in the release of polyphenols (Kumar et al., 2021).

Microwave assisted extraction: Microwave assisted extraction is a technique that involves heating the solvent in the vicinity of sample containing pineapple waste and employing microwave energy to extract polyphenols (Salve and Ray, 2020). The principle behind the extraction process is to use a microwave to generate heat, concisely electromagnetic energy is transformed into heat, accompanied by ionic conduction and molecular dipole rotation. Three successive processes involve the MAE mechanism: 1. solute separation from the sample matrix's active sites under increasing pressure and temperature; 2. solvent diffusion over the sample matrix; and 3. solute release from sample matrix to solvent (Salve and Ray, 2020). DESs (choline chloride-glycerol and choline chloride-malic acid), environmentally friendly solvents due to their low toxicity and high capacity for the extraction of polyphenols were used for the microwave assisted extraction of phenolic compounds from pineapple peels (Vargas-Serna et al., 2022).

Autohydrolysis

The polyphenols can be extracted from pineapple waste using autohydrolysis method which is a green method that utilises water as a solvent. Pineapple waste and distilled water were combined at different mass-to-volume ratios in steel cylinders immersed in a temperature-controlled oil bath. At the completion of the reaction period, the cylinders were immersed in an ice bath to cool down. Following, solid and liquid fractions were separated through filtration to produce hydrolysates. UHPLC was used to determine the amount of polyphenols present in the hydrolysates from the autohydrolysis experiment (Sepúlveda et al., 2018). Pineapple peel contains ferulic acid (FA), a phenolic antioxidant (Tilay et al., 2008) that also acts as a precursor for the production of vanillic acid and vanillin (Lun et al., 2014). Ferulic acid can be extracted from pineapple core waste through fermentation (Li et al., 2014). Vanillic acid and vanillin were produced by *Aspergillus niger* and *Pycnoporus cinnabarinus* using pineapple cannery waste as a substrate (Lun et al., 2014). The biotransformation of ferulic acid to vanillin occurs in a two-step process, the first step involved the production of vanillic acid from ferulic acid through the process of metabolism by the ascomycete, *Aspergillus niger* and then vanillin was obtained from vanillic acid by the basidiomycete, *Pycnoporus cinnabarinus* (Lesage-Meessen et al., 1996). This is a simple extraction method as compared to enzymatic (Faulds and Williamson, 1993) and alkaline extraction (Liu et al., 2006) methods which were cumbersome and expensive. In order to extract FA by alkali extraction, first the pineapple waste was crushed to powder and then a simple extraction method was employed. Resulting sample was taken into an Erlenmeyer flask and was saponified with NaOH followed by the addition of sodium hydrogen sulfite. Further, the mixture was centrifuged, and the supernatant was acidified with dilute HCl and the phenolic acids were extracted using ethyl acetate (Tilay et al., 2008).

2.1.5 Lipids

Pineapple pulp has been employed as a cheap and efficient carbon source for growth and lipid production by *Rhodotorula glutinis*. The sugars in the pineapple pulp were converted into lipids by *R. glutinis*. Liquid-liquid extraction utilizing chloroform and methanol as the extracting solvent was used as the standard procedure (Tinoi and

Rakariyatham, 2016). The primary components were discovered to be C-16 and C-18 fatty acids, which included linoleic acid, oleic acid, stearic acid, and palmitic acid (Figure 2.3) (Tinoi and Rakariyatham, 2016).

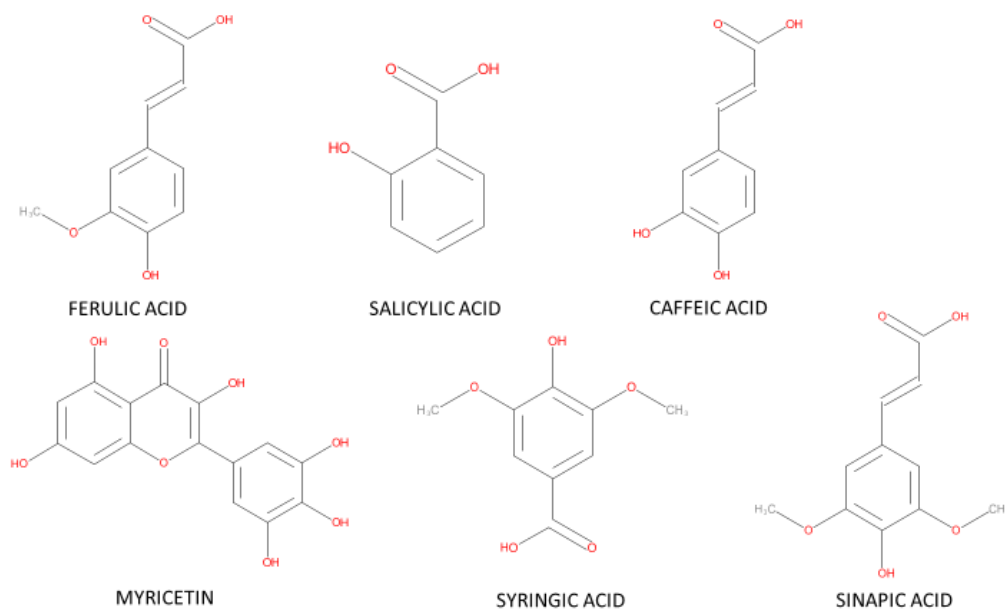


Figure 2.2: Structure of polyphenols derived from pineapple waste

2.1.6 Bromelain

The enzyme bromelain (Figure 2.4) and other cysteine proteases are found in various parts (Ketnawa et al., 2010; Rolle, 1998; Schieber et al., 2001) of pineapple waste (Nor et al., 2016). Reverse micellar system, aqueous two-phase extraction, cation exchange chromatography, and ammonium sulphate precipitation are common methods used for the extraction of bromelain from crude pineapple extract (Nadzirah et al., 2013). Bromelain was precipitated from pineapple fruit juice by adding ammonium sulphate $[(\text{NH}_4)_2\text{SO}_4]$ gradually with continuous stirring at a temperature of 4 °C (Devakate et al., 2009). Extraction of bromelain was done from pineapple peel by using the reverse micelles method (Wan et al., 2016). The reverse micelle was prepared in known proportions of n-hexane, 1-hexanol, surfactant and water with 1-hexanol and n-hexane taken in volume ratio of 1:9.

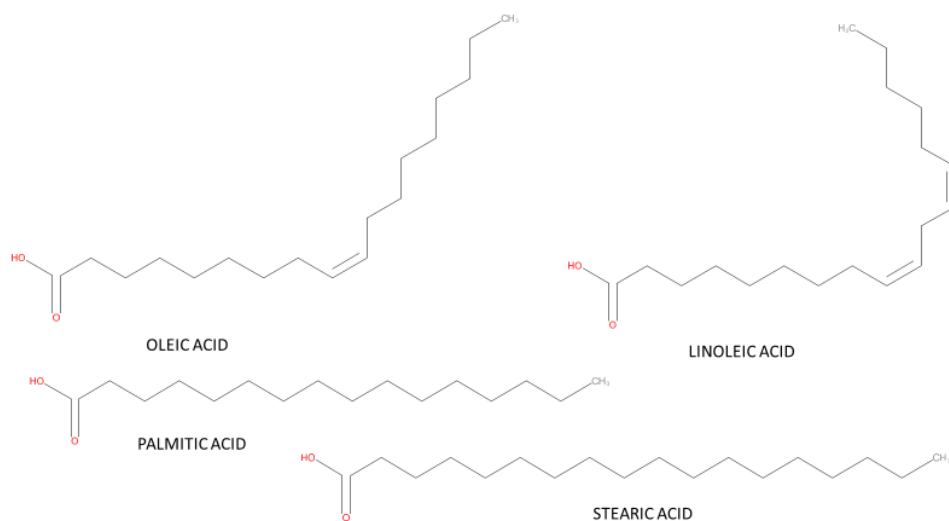


Figure 2.3: Structure of lipids derived from pineapple waste

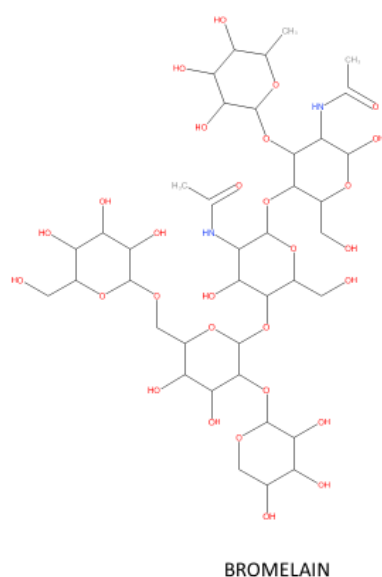


Figure 2.4: Structure of bromelain derived from pineapple waste

Phase separation was achieved by centrifuging for 30 to 50 minutes at 14,000 rpm. The aqueous phase was examined for bromelain activity after the reverse extraction (Wan et al., 2016). Aqueous two-phase system (ATPS) was employed for the extraction of bromelain from pineapple peels (Ketnawa et al., 2011). Bromelain was primarily partitioned to the phase that was high in polyethylene glycol (Ketnawa et al., 2011). Purified bromelain extracts were produced using consecutive batch

membrane processing techniques that included microfiltration, ultrafiltration, and ultracentrifugation (Doko et al., 1991).

2.1.7 Aromatic compounds

Pineapple crown leaves were subjected to pyrolysis that yielded oxygenated chemicals which were then transformed into aromatic compounds using the HZSM-5 catalyst at 600 °C. Fast pyrolysis, a high-temperature process was used that involved heating of PCL without oxygen to produce vapours, which were then instantly cooled to generate pyrolysis/bio-oil and which when passed over zeolites was changed into aromatic compounds (Peacocke and Bridgwater, 2000; Maneffa et al., 2016). The process began with the formation of anhydrous sugars from cellulose or hemicellulose molecules, which were then dehydrated to form furans and further reactions occurred on acid zeolite to give aromatic compounds (Barbosa et al., 2019; Maneffa et al., 2016). Through the process of fast catalytic pyrolysis, lignocellulosic biomass is converted into aromatic compounds of industrial interest like benzene, toluene, ethylbenzene, and xylene (Barbosa et al., 2019; Maneffa et al., 2016)

Table 1.2: Molecules and method of extraction

S. No.	Molecules extracted	Method of extraction and a brief description	Extraction time	Solvent	Advantages	Disadvantages	Reference
1.	Citric acid	Solid state fermentation (SSF) of glucose or sucrose by <i>Aspergillus niger</i> .	24 h	Methanol	Low cost, higher product stability	Growth rate of microbes on solid substratum is relatively slow, production of excessive heat in the medium	Ayeni et al., 2019; Papagianni, 2007
2.	Succinic acid	Fermentation of pineapple hydrosylate using <i>Actinobacillus succinogenes</i> , <i>Escherichia coli</i>	3-4 days	Sulfuric acid	Simple process, higher product stability	Time consuming, high cost	Jusoh et al., 2014; Pathanibul and Hongkulsup, 2021 Dessie et al., 2018
3.	Lactic acid	Fermentation using <i>Lactobacillus lactis</i> and the enzyme invertase to hydrolyze sucrose into glucose and fructose	72 h	Calcium alginate	Simple technology, higher yield, low cost	Difficult to scale up and maintain parameters like pH, moisture and temperature	Datta and Tsai, 1997; Idris and Suzana, 2006; Jin et al., 2005; Ueno et al., 2003
4.	Acetic acid	Fermentation using <i>Saccharomyces cerevisiae</i> , <i>Acetobacter aceti</i>	10-30 days	Ethanol	Simple process, higher product stability	Time consuming, high cost	Umaru, 2015; Roda et al., 2017

5.	Oxalic acid	Fermentation using <i>Aspergillus niger</i>	9 days	KH ₂ PO ₄ , MgSO ₄ and NaNO ₃	Simple technology, higher yield, low cost	Time consuming	Amenaghawon and Kazeem, 2020
6.	Essential oils	Hydro-distillation (HD) in which aqueous solution of pineapple peels was first added to a round bottom flask and then connected to cleverger-type apparatus	3 h	water	Simple instrumentation, low cost, free from organic solvents	Thermal degradation of products, high consumption of energy	Mohamad et al., 2019
		Hydro-distillation with enzyme-assisted pre-treatment (HDEA) in which pineapple peels were mixed with acetate buffer and acremonium cellulase solution for different enzyme loadings followed by HD procedure.	90 mins	acetate buffer	High efficiency, requires mild conditions	Only produced hydrosols instead of essential oil, expensive	Mohamad et al., 2019
		Supercritical fluid extraction (SFE) in which pineapple peels were placed in the SC-CO ₂ extraction apparatus	3 h	CO ₂	Solvent CO ₂ is inexpensive, solvent recycling can be done	Loss of desired compounds with improper solvent selection, specialized instrument required	Mohamad et al., 2019; Kamali et al., 2015

		followed by dynamic extraction					
7.	Carbohydrates	Enzymatic hydrolysis of hemicellulose to produce XOS	16 h	Acetate buffer	Able to produce phases which aren't stable at higher temperatures safely	Higher incubation temperatures can lead to disintegration of the hemicellulose	Banerjee et al., 2019
8.	Pectin	Acid extraction and ethanol precipitation in which dried PW was taken in a beaker followed by the addition of distilled water and sulphuric acid, then ethanol was added for the coagulation of filtrate	65 mins	Ethanol	Simple and requires manual work	High consumption of energy and material	Sarangi et al., 2020
		Ultrasound assisted extraction (UAE) which induced the collapse of cavitation bubbles near cell walls which causes cell disruption, thus causing stronger and enhanced solvent entrance into the cells and amplification of the mass transfer	15-30 mins	Acidified water	High yield, less solvent, green method	Requires optimisation	Shivamathi et al., 2022

		Microwave heating in which there is absorption of strong microwave energy to heat the solvent in order to permit the partitioning of analytes in the sample matrix into the solvent	1-20 minute	0.5 N H ₂ SO ₄ with pH 1.83 and 1:30 (w/v) solid-to-solvent ratio	Rapid, high yield with enhanced purity	High maintenance cost	Zakaria et al., 2021
9.	Starch	Mechanical extraction, a simple process of wet grinding and washing	48-50 h	Distilled water	Cost effective, takes less time	Choice of sample can be difficult	Nakthong et al., 2017
10.	Glucose	Enzymatic hydrolysis using cellulase	72 h	Sodium citrate buffer	Low toxicity	Time consuming	Banerjee et al., 2022; Mund et al., 2021
11.	Poly-phenols	Soxhlet extraction/solid-liquid extraction technique for polyphenols using soxhlet apparatus	6-8 h	Aqueous methanol	A very simple process that involves limited preparation	Time consuming, large volume of solvent required	Madhumeena et al., 2021; Salve and Ray, 2020
		Supercritical fluid extraction (SFE), a green technology due to use of supercritical CO ₂	10-60 mins	CO ₂	Takes less time and is eco safe, recycling of supercritical fluid is feasible.	Many parameters to optimize, CO ₂ is unsuitable for polar materials	Salve and Ray, 2020

		Pressurised solvent extraction in which analyte extraction from the sample is done under elevated temperature and pressure conditions.	5-10 mins	Organic solvents such as diethyl ether	Less extraction time, and less solvent required.	Cost of equipment and thermal degradation	Kaufmann and Christen, 2002
		Ultrasonic-assisted extraction method to extract polyphenols whose main principle involved acoustic cavitation	10–90 mins	Ethanol	Less solvent required and less extraction time, cost-effective	Requires optimisation, possible negative pressure cavitation	Carreira-Casais et al., 2021; Kumar et al., 2021; Salve and Ray, 2020
		Microwave assisted extraction in which electromagnetic energy is transformed into heat, accompanied by ionic conduction and molecular dipole rotation	30-60 sec	Ethanol, methanol, DESs	Decreased thermal gradients, equipment size reduced, rapid	Extraction solvent must absorb microwave energy	Alias and Abbas, 2017; Salve and Ray, 2020; Vargas-Serna et al., 2022
		Autohydrolysis, a green method that utilises only water as a solvent system	30 mins	Water	Absence of chemical solvent, no corrosion problems, simple to operate, cost-effective and economical	Less recovery of polyphenols	Batalha et al., 2015; Sepúlveda et al., 2018

12.	Ferulic acid	Enzymatic that includes microbial fermentation by feruloyl esterase	3 h	Dry methanol	Highly efficient	Lengthy, cumbersome, and uneconomical	Faulds and Williamson, 1993
		Alkaline, a simple extraction method was employed using alkaline hydrolysate	24 h	NaOH	Facile instrumentation	Not an environment friendly technique	Liu et al., 2006
13.	Lipids	Liquid-liquid extraction in which the sugars in pineapple pulp were converted into lipids by <i>R. glutinis</i>	4-5 hour	Chloroform, methanol	Useful to separate single component from multi-component mixture	Large amounts of solvents may be consumed	Tinoi and Rakariyatham, 2016
14.	Bromelain	Ammonium sulphate precipitation in which bromelain was precipitated from clarified pineapple extract by adding $[(\text{NH}_4)_2\text{SO}_4]$	15 min	–	Allows for the rapid, bulk precipitation of cellular proteins.	Bromelain produced is not as pure as expected causing another purification process to take place.	Devakate et al., 2009
		Reverse micellar system in which phase separation followed by reverse extraction; the aqueous phase was examined for bromelain.	60-90 min	1- hexanol to n-hexane in 1:9 volume ratio	Highly purified bromelain can be extracted.	Drop-in activity due to alteration in structures of proteins, slow rate of reverse extraction	Wan et al., 2016

		Aqueous two-phase extraction that was performed in 50-mL centrifuge tubes and the effect of PEG on the partitioning of bromelain was done	20-25min	17% MgSO ₄	simple, fast, and relatively cheap	Predicting the partitioning of biomolecules in ATPS can be difficult.	Ketnawa et al., 2011
15.	Aromatic compounds	Catalytic fast pyrolysis, a high-temperature process that involves rapid heating without oxygen	2-10 sec	Zeolite catalysts	Simple, inexpensive	Energy intensive, produces undesirable by-products	Barbosa et al., 2019; Peacocke and Bridgwater, 2000; Maneffa et al., 2016

2.2 VALUE-ADDITION TO PINEAPPLE WASTE

2.2.1 *Aerogels*

Pineapple fibres from pineapple leaves were explored as a viable source for cellulose-based aerogel development due to their biodegradability, low cost, high cellulose content, and exceptional mechanical properties. A highly porous cellulose aerogel with a high adsorption capacity can be produced by combining natural fibres from agricultural waste such as pineapple fibres with an adhesive reagent. Using deionized water as a solvent and polyvinyl alcohol (PVA) as an adhesive, flexible cellulose aerogel from raw pineapple-leaf fibres (PALF) have been produced (Do et al., 2020).

2.2.2 *Pineapple waste assisted nanoparticles synthesis*

Pineapple remnants including pericarp, fibres, leaves etc. contains high phenolic content, different kinds of phytochemicals and a high concentration of cellulose, hemicellulose, and lignin. All these properties of pineapple waste can be utilised in the production of nanoparticles including ZnO nanoparticles, carbon nanosheets, silver nanoparticles and nanocellulose.

ZnO nanoparticles

Pineapple plant extracts are rich in phytochemicals such as polyphenols, terpenoids, flavonoid alkaloids and sugars that function either as reducing agent or as capping or stabilising agent in the synthesis of nanoparticles. In the production of ZnO nanoparticles, pineapple peel extract was used as the capping agent and KOH solution and wood ash lye were utilised as reducing agents (Klinbumrung et al., 2022). The discarded pineapple pericarp can also be used to produce nanoparticles using sonication. Pineapple rind is a waste product that can be used as coating and stabilising agents in the production of nanoparticles (Mirgane et al., 2021).

Carbon nanosheets

Pineapple leaves, a novel sustainable source, were effectively used to prepare activated carbon in the form of extremely porous, open-channel carbon nanosheets (Sodtipinta et al., 2017). The process starts with the raw material being transformed

into pineapple leaf fibre (PALF) which is then used to produce activated carbon using hydrothermal process, chemical activation, and heat treatment in an argon atmosphere (Sodtipinta et al., 2017).

Silver nanoparticles (AgNPs)

Pineapple leaf was utilised to produce silver nanoparticles (Emeka et al., 2014). The pineapple peel extract acts as the reducing agent to produce silver nanoparticles by a cost-effective and ecologically friendly method of biosynthesis with size ranging from 20 nm to 30 nm (Syed Omar and Mustapa, 2018; Poadang et al., 2017). Pineapple leaf extract was used to produce AgNPs at a temperature of 70 °C. The smallest AgNPs particle size was 30 nm, which was attained in a solution of 1 mM AgNO₃ after 24 hours of stirring at 70 °C. The stability of the colloidal AgNPs lasted up to 7 days (Hamdiani and Shih, 2021). Without the use of any reducing or stabilising agents, an aqueous extract of pineapple peel waste was successfully used to synthesise ultra-small (average size 14–20 nm) AgNPs. Two key variables, namely the concentration ratio of peel extract to silver ion precursor and the synthesis pH, were discovered to be important for producing monodispersed and stable AgNPs (Agnihotri et al., 2018). Colloidal silver nanoparticles (AgNPs) were synthesised using pineapple peel extract as a reductant and stabiliser. The synthesised AgNPs had diameters ranging from 10 to 55 nm and were virtually spherical in shape. Phytochemical in pineapple peel extract were found responsible for the reduction and stabilisation of the biogenic AgNPs (Poadang et al., 2017).

Cellulose nanocrystals

There are three methods namely acid hydrolysis, enzymatic hydrolysis, and mechanical method for the extraction of nanocellulose (Nguyen et al., 2021). The pineapple crown is a rich source of cellulose and cellulose nanocrystals can be extracted from it by mercerization, bleaching and acid hydrolysis with H₂SO₄ (Prado and Spinacé, 2019). With the help of sulfuric acid hydrolysis, bleaching and alkali treatments, needle-like nanocellulose (PPNc) were extracted from pineapple peels (Dai et al., 2018). Pineapple peel residual juice contains sugar and nitrogen components that were helpful for the production of bacterial cellulose by

Gluconacetobacter xylinus, which upon sulfuric acid hydrolysis gave cellulose nanocrystals (CNC) (Anwar et al., 2015). The effective extraction of cellulose nanofibrils from pineapple leaf fibres using mechanical methods involves steam explosion method. In order to depolymerize and defibrillate pineapple leaf fibres and make nanofibrils, steam-coupled acid treatment is useful (Cherian et al., 2010). The steam explosion is characterised by high-pressure steaming and quick decompression. The material is saturated with steam at high pressure and temperature and following the pressure is suddenly released causing the water to flash evaporate and create a thermomechanical force that causes the material to rupture (Cherian et al., 2010). The pre-treatment method of steam explosion can be utilised to lower the amount of non-cellulosic components followed by acid treatment to give the nanocellulose (Cherian et al., 2010). Chemo-mechanical approach to extract nanocellulose from PALF (pineapple leaf fibres) involves chemical processing, sonication and milling. The pre-treatment aims to get rid of components like lignin and hemicellulose and further acid hydrolysis is used to obtain nanocellulose (Gadzama et al., 2020) (Table 2.2).

Table 2.3: Extraction techniques of cellulose nanocrystals

Cellulose nano-crystals	Acid hydrolysis	The needle-like nano cellulose (PPNc), isolated using sulfuric acid hydrolysis	15 h	Sulfuric acid	Simple waste processing and low raw material costs.	Long reaction time and high concentrations of acid results in corrosion	Anwar et al., 2015; Dai et al., 2018; Prado and Spinacé, 2019
	Enzymatic hydrolysis	Production of nanocellulose by enzymes of various activity	72 h	Sodium-citrate buffer	Does not release toxic residues and can be conducted under room temperature and pressure for energy-saving purposes	Less effective than acid hydrolysis	de Campos et al., 2013
	Steam explosion method	Extraction of cellulose nanofibrils from pineapple leaf fibres by steam coupled acid treatment	6-7 h	Oxalic acid	Environmentally friendly and requires low capital investment	Some changes occur in the arrangement of macromolecular chains and degradation of cellulose, hemicelluloses and lignin is difficult to control.	Cherian et al., 2010
	Chemo-mechanical	Extraction using pre-treatment employing bleaching, acid hydrolysis followed by sonication and milling.	5-6 h	NaOH	Reduced need for extensive chemical pre-treatment, minimized the use of expensive and potentially environmentally damaging chemicals	Time consuming	Gadzama et al., 2020

2.2.3 *Fibres and polymer matrix composites*

One of the widely accessible waste materials from pineapple plant is pineapple leaf fibre (PALF). Ash (1.1%), lignin (5–12%), and holocellulose (70–82%) make up the chemical composition of PALF (Arib et al., 2004; Asim et al., 2015). PALF have been used as raw materials for the industries rather than the costly and non-renewable synthetic fibre. Natural fibre's only disadvantage is its tendency to absorb moisture which can be overcome by adding chemicals to alter its surface property (Asim et al., 2015). Manual or hand scraping, mechanical decortication, and retting are some of the conventional techniques for extracting PALF. These techniques produce PALF in the form of lengthy fibre bundles. PALF can be further processed using sodium hydroxide and the mechanical forces of a high-speed mixer to create pineapple leaf microfiber (PALMF). Both PALF and PALMF have a strong potential as reinforcement for polymer matrix composites due to their good mechanical capabilities (Kengkhetkit et al., 2018). Low-density polyethylene (LDPE) composites (George et al., 1998), biodegradable plastic composites (Asim et al., 2015), and reinforced polymer composites (Mishra et al., 2001; Pavithran et al., 1987) using pineapple (PALF) showed excellent mechanical qualities.

2.2.4 *Anti-dyeing agent*

The waste from pineapple such as the peel, core and crown has also been used to remove dyes as pineapple adsorbent is inexpensive and can reduce disposal costs (Subki, 2017). The magnetized activated carbon from pineapple crown leaves was prepared to be used as an adsorbent for removing methyl violet, an organic dye that is commonly used in the paint and textile industries (Astuti et al., 2019), mesoporous activated carbons were produced from PCL to remove caffeine (Beltrame et al., 2018) and titanium dioxide nano bio-adsorbent (TiO₂L) was also synthesised to remove Victoria blue dye (Fakhar et al., 2020). Pineapple stem was employed as an adsorbent to remove the cationic dye (Hameed et al., 2009) and safranin-O from water (Mohammed et al., 2014) and to extract methylene blue from an aqueous solution. Biochar and activated carbon were produced from pineapple waste biomass (PWB) for the purpose of immobilizing lipase and RBBR (remazol brilliant blue R) dye

adsorption (Veeramalai et al., 2022). Pineapple Peels were used for the biosorption of an anionic dye, Eosin Yellow (Ugbe et al., 2018).

2.2.5 For heavy metal intoxication

Pineapple waste have attracted attention as sorbents for hazardous metals. Heavy metals and chemical oxygen demand (COD) in wastewater are reduced by activated carbon made from pineapple waste (Subki, 2017). Pineapple fruit peel biomass that has undergone chemical oxidation has been employed as a biosorbent to remove Cd(II) and Pb(II) from aqueous solutions (Ahmad et al., 2016). The pineapple crown leaf was employed as a bioadsorbent of Cr(VI) and Cr(III) in the aqueous phase, an affordable approach for the treatment of wastewater (Gogoi et al., 2018; Ponou et al., 2011). In order to remove heavy metals (cadmium, lead, and zinc) from contaminated soils, pineapple waste is also employed as a soil remediation agent (Feng et al., 2018). The stem of the pineapple plant is employed as an adsorbent to lower the concentration of Pb (II) in aqueous solutions (Ayob et al., 2020; Ting et al., 2019). Cell-EDTA and Cell-CM, produced either by the modification of cellulose fibre obtained from pineapple leaves with ethylenediaminetetraacetic acid (EDTA) and carboxymethyl (CM) groups respectively were used as heavy metal ion adsorbents of Pb(II) and Cd(II) (Daochalermwong et al., 2020) or by the reaction with succinic anhydride in pyridine and dimethyl sulfoxide solvent or by chemical modification, i.e., the introduction of carboxylic groups were used as heavy metal ion adsorbents of Cu(II), Cd(II), and Pb(II) (Hu and Huang, 2010). The adsorption of cadmium and lead ions is more effective on adsorbents made from pineapple waste after treatment with sodium hydroxide (Mopoung and Kengkhetkit, 2016). For removing Fe(III) from aqueous solutions, KMnO₄ modified carbon was prepared from the pineapple leaf fibre (Mopoung and Bunterm, 2016). For the elimination of Cr(VI) from the aqueous solution, pineapple leaf fibre was surface-modified by alkali solution (APF), followed by polyethyleneimine-grafting utilising carbamate-linkage reaction (APF-PEI) (Tangtubtim and Saikrasun, 2019).

2.2.6 *Pineapple waste as carbon source*

Bioenergy

Pineapple waste has been utilised as a fuel and using zinc and copper electrodes, this produced bioelectricity using single-chamber microbial fuel cells (Rojas-Flores et al., 2022). Waste from pineapples including peel, core, leaf and crown have been utilised to produce biofuels like biohydrogen, bioethanol, biodiesel, biobutanol, biogas and biohythane which are environmentally beneficial fuels and are the energy carriers to meet future needs.

Production of biohydrogen

Waste from pineapples (peel, core, and leaf) has been utilised in the production of biohydrogen (Cahyari et al., 2018). Biohydrogen is the preferred energy carrier to meet future needs due to its better properties such as lower emissions of greenhouse gases, high energy density, high energy conversion rate using a fuel cell (Cahyari et al., 2018) and use of less energy (Reungsang and Sreela-or, 2013). Pineapple waste were fermented in dark, mesophilic environment to produce biohydrogen (Cahyari et al., 2018). The fermentation process has a significant potential for commercialization energy production in future because it can be carried out at standard pressure and temperature conditions (Abd Jalil et al., 2018). Anaerobic microorganisms were used in fermentation due to their ability to utilise carbon substrates and to release hydrogen (Sivagurunathan et al., 2017). The well-known bacteria *Clostridium* sp. was found to be ideal for higher yields of biohydrogen (Bru et al., 2012). Co-culture approach for biohydrogen production was found to be the ideal choice as it resulted in enhancement of cell activity and culturing in order to increase production in a short period of fermentation (Goers et al., 2014; Kao et al., 2014).

Production of bioethanol

The high levels of sugar and lignocellulose found in the peel and crown of PW proved to be a valuable source for fermentation in the production of bioethanol (Casabar et al., 2019; Tropea et al., 2014). Three different methods were used for the extraction of bioethanol from PW: simultaneous saccharification and fermentation

(SSF), separate hydrolysis and fermentation (SHF), and direct fermentation (DF) (Casabar et al., 2019). Pineapple waste can be utilised in the production of cellulase using *Trichoderma reesei* where the cellulase produced was used in the production of bioethanol (Saravanan et al., 2013).

Production of biodiesel

Pineapple leaves were converted into biodiesel through the process of transesterification and calcination. The calcined pineapple leaves (CPL) exhibited a high catalytic efficiency and conversions greater than 98% were measured. CPL were found to reduce biowaste while acting as a highly effective biodiesel catalyst (Barros et al., 2020). Pineapple waste was used as a carbon substrate for the production of lipids using *Candida tropicalis* as the microorganism through the process of fermentation. Extracted lipids were then converted into fatty acids methyl esters (FAME) called the biodiesel through the transesterification reaction (Kanakdande et al., 2020). Pineapple peels were used to produce lipase by anaerobic fermentation and were found to be a prospective choice because of non-toxicity, tolerance to organic solvents, reusability, and insensitivity to free fatty acids. Partially purified lipase (PPL) could be an efficient and effective biocatalyst for biodiesel production as the lipase generated was found to exhibit greater catalytic efficiency for the conversion of palm oil into biodiesel (Selvakumar and Sivashanmugam, 2017).

Production of biobutanol

ABE fermentation, also known as acetone-butanol-ethanol synthesis is the method used to produce biobutanol from pineapple waste. *Clostridium* sp. converts sugars present in pineapple waste to butanol by employing acid hydrolysis and detoxification with activated carbon (Huzir et al., 2018; Khedkar et al., 2017). *C. beijerinckii* (Sanguanchaipaiwong and Leksawasdi, 2018) and *C. acetobutylicum* (Khedkar et al., 2017) are the two most commonly used solventogenic species for the synthesis of butanol.

Production of biogas

Pineapple peels were found to be a potential source for the production of biogas due to their high cellulose, hemicellulose, and other carbohydrate content. Mixed fruit wastes containing pineapple waste was used for biogas production (Lane, 1984; Viswanath et al., 1992). Under varied conditions, pineapple peels produced biogas that ranged from 0.41 to 0.67 mg/kg in volatile solids with a methane content of 41 to 65 % (Rani and Nand, 2004). By adopting semicontinuous anaerobic digestion, pineapple waste was used to produce methane, with a maximum methane concentration of 51% and a maximum daily biogas production rate of up to 1682 mL/day (Bardiya et al., 1996). Using municipal sewage sludge as the H₂ producer, pineapple waste was used as the carbon source (Wang et al., 2006). Volatile fatty acids and methane both were produced using solid pineapple waste. Along with methane, acetic, propionic, butyric, isobutyric, and valeric acids were also produced (Babel et al., 2004). The nutritional requirement for economically viable biohydrogen synthesis was obtained by co-digestion of fruit pineapple waste (Dareioti and Kornaros, 2015; Sen et al., 2016).

Single cell protein (SCP)

Single cell protein refers to the dried, dead microbial cells or total amount of protein that can be isolated from pure microbial cultures of filamentous fungi, algae, bacteria, unicellular algae, and cyanobacteria. These microbial cultures are grown on diverse carbon sources such as pineapple waste and utilized as a protein supplement (Mensah and Twumasi, 2017). Pineapple waste served as the carbon source for the fermentation media on which the two strains of yeast, *Saccharomyces cerevisiae* and *Candida tropicalis* were cultivated. Using conventional methods, PW and other food wastes were used as the growing medium for *A. niger* (Oshoma and Eguakun-Owie, 2018). The biomass yield and protein synthesis within yeast cells were improved by using pineapple waste as a substrate (Dharumadurai et al., 2011).

Enzyme production

β -Glucosidase enzyme was produced from pineapple waste by *P. miczynskii* (Beitel and Knob, 2013). Esterases, a significant class of hydrolytic enzymes was also produced from pineapple waste enriched soil using esterase producing bacteria,

Bacillus subtilis E9 and *Bacillus* sp. E46 (Soumya and Kochupurackal, 2020). PW was employed as nutrient substance in culture broth and cellulase production. It was used as the substrate for optimisation of cellulase production utilising statistical methods based on experimental designs using *Trichoderma reesei*. Fermentation process was carried out in Erlenmeyer flasks with pineapple waste powder. Flask was then covered with cotton, and it was autoclaved for 20 min. The flasks were then kept at room temperature for 48 hours post cooling. Cellulase production was determined to be at its highest on the sixth day of fermentation (Saravanan et al., 2013).

2.2.7 *Therapeutic applications*

Pineapple peel has the potential for medical usage, including source for the synthesis of antibiotics (Lubaina et al., 2019). Pineapple peel extract of the fermented fruit can prove to be an emerging potential in therapeutic approaches for cancer (Rashad et al., 2015). Transcinnamic acid and ferulic acid work together to give the plant its antibacterial, anticancer, and antioxidant effects (Jayashree et al., 2017) and gallic acid also contributes to a variety of characteristic properties that includes antibacterial, antifungal, and anticancer effects (Polanía et al., 2022). Pineapple waste contains polyphenols in its skin, pulp, and juice (Yahya et al., 2019). Polyphenols are known to exhibit biological and antioxidant activity (Segovia Gómez and Almajano Pablos, 2016). The two main types of polyphenols in pineapples found responsible for its antioxidant activity were flavonoids and phenolic acids (da Silva et al., 2013). For the recovery of polyphenols from dried PS, a more environmentally friendly ultrasound-assisted extraction (UAE) approach that requires less solvent and reduced extraction time (Zardo et al., 2019) was used (Yahya et al., 2019). A flavonoid known as myricetin is linked to antioxidant activity and is also involved in cellular processes that inhibit the growth of tumor cells (Johnson, 2004). Three phenolic acids found in pineapples—caffeic acid, p-coumaric acid, and ferulic acid—were found to exhibit antioxidant, anticancer, and antibacterial activity (Heleno et al., 2015). The total phenolic content and antioxidant activities of methanolic extracts of pineapple residues (pulp, seeds, and peels) were determined, utilising DPPH and superoxide anion scavenging activity (de Oliveira et al., 2009; Li et al., 2014). Catechin, epicatechin, gallic acid, and ferulic acid were discovered to be the main polyphenolic components

present in pineapple peels and these components were responsible for its antioxidant activity (Li et al., 2014). Raw pineapple extracts are rich in nutrients and bioactive substances like proteins, minerals, lipids, vitamin C, phenolic compounds, flavonoids, and carotenes and thus offers a variety of therapeutic benefits (Romelle et al., 2016; Niramol et al., 2018; Sah et al., 2016). Bioactive components from *Ananas comosus* reduced the release of inflammatory cytokines and decreased T-cell proliferation (Kumar et al., 2022). Consequently, *Ananas comosus* nanoemulsion (NE) can be utilised for the treatment of cancer (Kumar et al., 2022). PW waste has the potential to be used as a nutraceutical for the treatment of diabetes (Riya et al., 2014). The extraction is usually done by traditional methods such as macerating, hydro-distillation, and solid-liquid extraction utilising organic solvents (Giacometti et al., 2018; Wang and Weller, 2006).

The potential of pineapple peel in growth medium for the *Lactobacillus* species was also explored. Pineapple waste was used as the source of carbon for the fermentation media to support the growth of three probiotic strains of *Lactobacillus* sp. (Pyar et al., 2014). Pineapple peel was found to be an essential prebiotic component in the production of yogurt with higher nutrient content and functionality (Sah et al., 2015). Pineapple peel flour contains around 60% of fibre and 20% of the total amount of soluble carbohydrates which makes the pineapple peel a rich carbon source for the growth of lactic acid bacteria. Pineapple peel's fermentable properties, high level of total dietary fibre, and antioxidant characteristics makes it a functional ingredient (Diaz-Vela et al., 2013). PW has a high reducing sugar content (30.5 mg/100g) as well as a high ash level (1.8 mg/100 g). It also showed high concentrations of crude fibre, non-reducing sugars, carbohydrates, and proteins, all of which can act as substrate for the growth of probiotics and yeast fermentation which results in the production of ethanol and single-cell protein (Hemalatha and Anbuselvi, 2013). Additionally, pineapple peel and stem extracts were taken in account for their prebiotic action in the development of a new functional ingredient. Six different probiotic strains from the two unique genera *Lactobacillus* sp. and *Bifidobacterium* sp. were used for the initial screening. With the exception of *Lactobacillus acidophilus*, all other bacteria showed favourable growth in response to pineapple extracts (Campos, 2018).

Hydrogels

Hydrogels can be produced from the cellulose present in pineapple peels. To obtain CMC, carboxymethylation was carried out by the reaction of pineapple cellulose with chloroacetic acid (Tuyet Phan et al., 2021). Carboxymethyl cellulose (CMC) was obtained from pineapple leaf waste through the process of etherification by treating pineapple leaf powder with a solution of NaOH followed by HNO₃. The hydrogel was prepared by graft copolymerization of acrylic acid and acrylamide with the synthesised CMC (Tuyet Phan et al., 2021). N,N'-methylenebisacrylamide (MBA) acted as the crosslinking agent and ammonium persulfate (APS) as the initiator during the reaction (Tuyet Phan et al., 2021). By grafting acrylic acid and carrying out homogenous acetylation of cellulose in ionic liquid 1-butyl-3-methylimidazolium chloride, pineapple peel-based hydrogels and sepia ink were produced (Dai and Huang, 2016). First, pineapple pulp was filtered through etamine and the leftover material was dried before being pulverised through a mesh screen. Dried pineapple peel powder was incubated with distilled water for two hours to extract the cellulose. Sodium chlorite was combined with the insoluble residue to remove lignin after the filtration. The residue was then shaken with potassium hydroxide solution at the room temperature. PPC was obtained after the solution was cleaned and dried with water and ethanol. Superabsorbent hydrogels were synthesised by grafting acrylic acid and acrylamide onto carboxymethyl cellulose from pineapple peels (Dai and Huang, 2017). Carclazyte was additionally added to the hydrogels to enhance their pH sensitivity and swelling capacity.

2.2.8 Reformation of retrieved compounds into valuable products for use in food and drinks

The production of rock buns, a snack diet enhanced with pineapple pomace contains larger amounts of fibre and vitamin C which boosts the nutritional content, fosters dietary diversity and ensures food sustainability (Badjona et al., 2019).

Production of biopolymers

Pineapple waste, particularly the stem contains holocellulose and hemicellulose which makes it a suitable substrate for the production of biopolymers

(Chen, 2014). A natural polymer cellulose is obtained by refluxing the pineapple by-products (Xu et al., 2009). Carboxymethylcellulose (CMC), a biopolymer can be obtained from pineapple peel through cellulose by the process of etherification (Ramadhan and Handayani, 2020). Tapioca-based bioplastic resin (TBR) can be prepared from pineapple leaf fiber (PALF) (Mathivanan et al., 2016). *B.drentensis* strain BP17 is a bacteria used for PHB production using pineapple peel as a low-cost alternative carbon source (Penkhrue et al., 2020). For extracting biopolymers, the pineapple peels were fermented with ammonium sulphate or dipotassium phosphate, followed by hydrolysis with H_2SO_4 at a rate of 4000 rpm (Maraveas, 2020). Waste from the agro-industrial sector like pineapple peel residues can also be used to make biopolymers like polyhydroxyalkanoates (PHA) through fermentation using *Ralstonia eutropha* ATCC 17697 strain.

2.2.9 Animal Feed and organic fertilizers

Pineapple waste has also been utilised to produce high quality animal feed because of its palatability and presence of anti-nutrient factors like phytic acid and tannic acid (Bakri et al., 2020; Sruamsiri, 2007). The animal feed containing pineapple waste proved to boost the protein intake of animals and also increased the animal weight and digestibility (Costa et al., 2007). Pineapple liquid waste can be utilised as organic fertilizers (Sutanto, 2016) by using the process known as vermicomposting in which PLW acts as a substrate for the growth of bacteria. It decomposes very rapidly due to its high moisture content and presence of soluble carbohydrates (Sruamsiri, 2007). The nutritional content of pineapple waste combined with rice straw can be preserved by the process of ensiling (Jitramano, 2005). Dried and ensiled pineapple waste can be used as extra roughage which can replace 50% of the roughage in the total mixed ration for dairy cattle without having an impact on animal productivity (Sruamsiri, 2007). The growth performance of *Nile tilapia* (an aquatic organism) was enhanced by feed formulation using waste from pineapples (Sukri et al., 2022). Ruminant feed made from the leaves and the outer peel of the pineapples can be used in canning (Tran, 2006). Pineapple pulp can be also utilised for human consumption (Cabrera et al., 2000). However, the high fiber content and low protein content of the

by-product of the pineapple processing sector makes it unattractive as animal feed (Correia et al., 2004).

Table 2.4: Summary of applications of pineapple waste

S. No.	Production	Part of pineapple used	Benefit of production	Reference
1.	Aerogels	Pineapple fibres	Ethylene gas adsorption and nickel (II) ion removal, Oil/water separation, Dye removal, Energy storage as supercapacitor electrode, Cr (VI) removal.	Do et al., 2022; Feng et al., 2015; Hao et al., 2014; Lim et al., 2020; Tan et al., 2023
2.	ZnO nanoparticles	Pineapple peel, pericarps	Antibacterial properties and capacity for wound healing, use in cotton textiles for ultraviolet light protection, wastewater treatment and photocatalytic activity.	Belay et al., 2020; Kaushik et al., 2019; Khalafi et al., 2019; Mirgane et al., 2021
3.	Carbon nanosheets	Pineapple leaves	High-performance energy storage devices like supercapacitors	Sodtipinta et al., 2017
4.	Silver nanoparticles	Pineapple leaves	Medicinal, cosmetic and environmental applications including the ability to eradicate harmful bacterial strains	Agnihotri et al., 2018
5.	Cellulose nanocrystals	Pineapple peel	Simple waste processing, and low raw material costs.	Dai et al., 2018
6.	Fibres	Pineapple leaf fiber (PALF)	Low-density polyethylene (LDPE) composites, biodegradable plastic composites and reinforced polymer composites	Asim et al., 2015; George et al., 1998; Mishra et al., 2001; Pavithran et al., 1987
7.	Anti-dyeing agent	Pineapple peel, core and crown	Paint and textile industries	Astuti et al., 2019

8.	Heavy metal intoxication	Pineapple fruit peel, crown leaf	Soil remediation, wastewater treatment	Feng et al., 2018
9.	Biohydrogen	Pineapple peel, core, and leaf	Energy carrier to meet future needs	Reungsang and Sreela-or, 2013
10.	Bioethanol	Pineapple peel, crown	Sustainable bioethanol production	Tropea et al., 2014
11.	Biodiesel	Pineapple leaves	Environmentally beneficial fuel	Barros et al., 2020
12.	Biobutanol	Pineapple peel	Sustainable biobutanol production	Huzir et al., 2018
13.	Biogas	Pineapple peel	Environmentally beneficial fuel	Rani and Nand, 2004
14.	Single cell protein	Pineapple skin waste, peels, mesocarp	Address the world's protein deficit	Dharumadurai et al., 2011
15.	Enzyme production	Pineapple pulp and peels	Food industries, biomass hydrolysis for bioethanol production and enhancing the flavor of wine, tea and fruit juice	Beitel and Knob, 2013
16.	Antioxidants	Skin, pulp, and juice	Exhibits antioxidant, anticancer, and antibacterial activity	Heleno et al., 2015
17.	Medicinal properties	Pineapple peel	Antibacterial, anticancer, and antioxidant properties	Jayashree et al., 2017
18.	Probiotics	Pineapple peel	Production of ethanol and single-cell protein	Hemalatha and Anbuselvi, 2013
19.	Hydrogels	Pineapple peel	Water-manageable materials, slowrelease fertiliser and controlled drug release, contact lenses, personal care items, and wound dressings	Caló and Khutoryanskiy, 2015; Dai and Huang, 2017
20.	Biopolymer s	Pineapple peel	Biomedical applications, including heart valves, tissue regeneration scaffolds, vascular grafts, stents, dental	Zinn et al., 2001

			and maxillofacial procedures, orthopedics, urology and wound management supplies (sutures and dressings).	
21.	Animal feed	Crown, core, peel, leaves, and leftover flesh trimmings	Animal feed and fertiliser	Sukri et al., 2022

CHAPTER - 3

CONCLUSION

The pineapple industry involves huge quantity of by-products (stems, roots, and leaves) that possesses high economic potential. Collaborative efforts were made to recover and utilise pineapple waste in production of important chemicals such as organic acids, essential oils, nanocellulose, along with their application in pharmaceuticals, cosmetic, and textile sectors.

This article covered the processing of pineapple waste into useful molecules and highlights its utility in various sectors of the community. Pineapple by-products have a great potential in the production of organic acids, polyphenols, bromelain, pectin, and holds applications in generation of bioenergy. Pineapple waste was utilised as fermentable substrate which served as both, a carbon source for acid fermentation and as medium for bacterial growth. Thus, it can be utilised to produce organic acids namely citric acid, succinic acid, lactic acid, acetic acid and oxalic acid through the process of fermentation by employing different microorganisms. Essential oil can also be extracted from the peel and fibre of pineapple waste. Furthermore, xylan-rich lignocellulosic waste from pineapples can be used to produce XOS. Pineapple skin contained enough amount of phenolic chemicals that can be utilised in the pharmaceutical and cosmetics sectors. Salicylic acid, caffeic acid, myricetin, syringic acid, sinapic acid, ferulic acid etc. were the identified phenolics in pineapple, that were responsible for its antioxidant activity. Pineapple peel exhibited strong potential as a natural plasticizer and a source of pectin. Bromelain, a proteolytic enzyme was extracted from pineapple waste that can be utilised in pharmaceutical industries. Additionally, dietary fibres and phenolic antioxidants could be employed as a future source of nutraceuticals, providing low-income areas with a significant low-cost nutritional dietary supplement. Pineapple remnants such as pericarp, fibres, leaves etc. contained high phenolic content, various phytochemicals and a high concentration of cellulose, hemicellulose, and lignin which lead to the production of nanoparticles like ZnO nanoparticles, carbon nanosheets, and silver nanoparticles. The pineapple plant's stems and leaves were a great source for white, creamy, silk-like fibre. Pineapple leaf fibre (PALF) can replace the costly and non-renewable synthetic fibre and thus can act as a new source of raw materials for the industries. Further the usage of PALF as the source for cellulose-based aerogel development can prove effective due to their biodegradability, low cost, exceptional mechanical properties and high cellulose

content. The peel, core, and crown, can also be used to remove dyes due to economic reasons and can also be utilised to produce biofuels like biohydrogen, bioethanol, biodiesel, biobutanol, biogas, biohythane which are environment friendly energy carriers to satisfy future needs. Pineapple waste have recently gained attention as sorbents for hazardous metals, due to their ability to adsorb toxic metals from aqueous systems. Pineapple peel was utilised as the source for the synthesis of antibiotics and also have the potential to be used as a nutraceutical in treatment of diabetes. Peels of pineapple can also be employed in making bioplastics (or biopolymers) by refluxing it with acid or alkaline solutions. Other such uses included utilising pineapple waste as fuel, in production of rock buns, and in making paper. Therefore, in conclusion, the waste from pineapples can be turned into valuable products if innovative scientific techniques are used.

CHAPTER - 4

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