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# REVIEW OF RICE BRAN WAX EXTRACTION AND REFINING PROCESS

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## ABSTRACT

Rice bran wax (RBW) is a valuable by-product obtained during the refining of rice bran oil which contains approximately 2-5% wax before refining, which makes it one of the oils with the highest wax content. Crude bran wax contains a significant amount of oil (up to 60%), and small amounts of impurities, water, gums, resinous matter, free fatty acid, and phospholipids. The presence of these impurities affects the color and performance of the product. Refining crude wax is necessary for its use in the industry. The wax extraction is accomplished by mechanical and chemical processes during the dewaxing of oil. It can be separated from oil by tank settling, winterization, batch chromatography, etc. Refining crude rice bran wax typically involves defatting, bleaching, filtration, and deodorization to ensure the high purity and quality of the resultant wax. Defatting can remove about 90% of the oil from the crude wax in the first extraction and up to 98% in subsequent extractions. Refining of crude rice bran wax is crucial in removing impurities such as free fatty acids, resinous matter, residual oil, and smell, only after which it can be used in food, cosmetics, and pharmaceutical industries. The crude wax can be used as such in products like shoe polishes where odor and color are of no great importance.

**KEYWORDS:** Rice bran wax, uses, extraction, refining.

## INTRODUCTION

In an increasingly resource-constrained era, using waste and by-products generated from grain processing has a wide appeal due to their nutritive value, economic aspect, and sustainability. The current trend of going natural, and organic has increased the appeal for better utilization of grain waste by converting them into value-added products following the principles of circular economy.

Rice bran oil is notable for its high content of antioxidants and wax. Rice grows in watered paddies and under strong sunshine, which can explain why rice bran, the outer layer of rice grain after the hull is removed by milling processes, contains higher wax and antioxidants than other edible oils. Rice bran wax (RBW), an intermediate by-product of rice bran oil refining industry, has been one of the most underutilized waste materials of the food processing industry. About 3.8 lakh tons of crude rice bran oil is processed in India every year. Dewaxing is an important step in oil refining. Rice bran oil has the highest wax content among vegetable oils and can reach up to 5%.[1] The crude wax separated from the oil retains about 25% or more amount of the oil and its production is estimated to be around 35,000 to 60,000 tons, which can be potentially used in a lot of industries viz paper coating, candles, waterproofing polish (floor, furniture, and shoe), carbon paper, printing inks, adhesives, fruit and vegetable coatings, cosmetics, pharmaceutical preparations, etc.[2] Purified rice bran wax can partially substitute other more expensive waxes of natural origin as well as petroleum-based waxes. The wax obtained from rice bran oil refining is presently underutilized for the want of suitable processing technologies.

Rice bran wax consists of high molecular weight monoesters. These are long-chain saturated C46-C62 esters from C20-C36 fatty alcohols and C20-C26 fatty acids. The major components of rice bran wax are aliphatic acids (wax acids) and higher alcohol esters along with free fatty acids (palmitic acid), squalene, and phospholipids. The aliphatic acids consist of palmitic acid (C16), behenic acid (C22), lignoceric acid (C24), and other higher wax acids. The higher alcohol esters consist mainly of ceryl alcohol (C26) and melissyl alcohol (C30).[3] The general characteristics of rice bran wax are: solid state, yellowish white color, bland taste, soluble in hot alcohol benzene, carbon tetrachloride, ether, chloroform, isopropyl ether and insoluble in water, melting point 75-82$^{o}$C, refractive index 1.4684-1.4686, iodine value 50-25, acid value 10-17, saponification value 73-89, unsaponifiable matter 48%. Because of its comparable spreadability, diffusability, and lesser water absorbing capacity characteristics, it can be used as a substitute for more expensive standard waxes in ointments[4] and moisturizers,[5] and as a substitute for beeswax and carnauba wax due to its similar properties.[6]

The rice grain obtained after threshing and milling still contains the germbud and are surrounded by silverskin which is further removed by mechanical processing to produce rice bran alongside the stripped rice. This rice bran comprises lipid fractions, primarily consisting of fatty oils and wax-like components. The wax is found in the oil obtained by pressing or solvent extraction, from which it can be further isolated by freezing as they are poorly Soluble at low temperatures.[7]

**RICE BRAN WAX EXTRACTION AND REFINING**

Rice bran crude wax has deep color and high pigment content, which seriously affects the appearance and performance of wax products and are two major obstacles hindering the use of RBW. Various RBW extraction and refining processes have been mentioned in great detail like tank settling, solvent extraction, cold and hot extraction, batch chromatography, mechanical extraction, fractionation, etc. Almost all of them use solvents for wax recovery, cold press dewaxing of rice oil for crude wax recovery, and bleaching and deodorization processes for further refining of wax. The solvent extraction method utilizes the difference in solubility between wax and oil in organic solvents to separate them, and this method achieves the effect of removing part of phospholipids at the same time as extracting refined rice bran wax. Crude bran wax contains small amounts of mechanical impurities, water, gums, and phospholipids. The presence of these impurities affects the color and performance of the product. The main factors affecting oil removal include solvent type, solvent ratio, extraction temperature, extraction time, cooling temperature, etc.[8]

Wax can be separated from Rice bran oil by gravity settling followed by decanting. The oil is gradually allowed to cool for wax crystallization followed by filtration or centrifugation to recover the wax sludge. The wax content of crude oil depends on the variety of rice, milling technique, method of oil extraction, and extraction temperature. Extraction temperature affects both the type of wax present and its quantity. For example, extraction at 50$^{o}$C yields two to three times more wax than extraction at 20$^{o}$C. Initial dewaxing may be gravity settling followed by decanting. The oil is gradually cooled to allow wax crystallization followed by filtration or centrifugation to recover the wax sludge.[9]

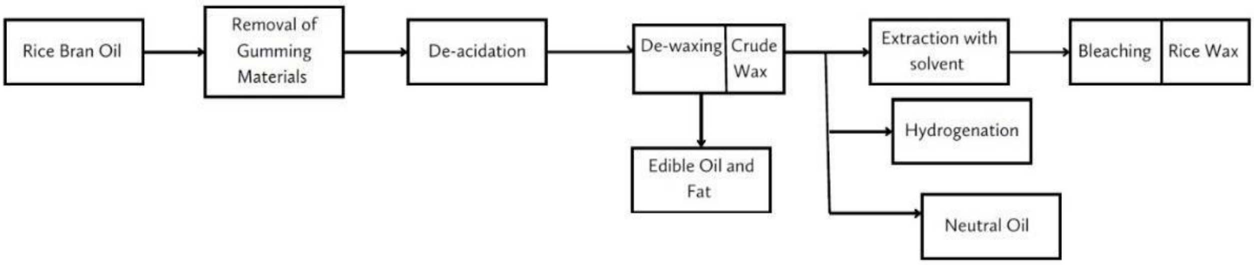
Vali proposed a two-step process for crude wax refining, that included defatting of crude rice bran wax and bleaching of the defatted wax. The defatting of the crude wax was achieved by dissolving the crude wax in hexane, the solution was then refluxed at 65$^{o}$C for 30 minutes. The contents were then cooled and insoluble wax was filtered off. The defatted wax was then dissolved in isopropanol and refluxed to remove impurities such as polar lipids, resinous matter, free fatty acid, and residual oil. The contents were cooled and the insoluble wax crystals were filtered off, which yielded a dark brown, powdery material on drying. The defatted wax contains a substantial amount of resinous matter which was then removed by bleaching. The bleaching was done by refluxing the wax with isopropanol and aqueous sodium borohydride (NaBH4) solution. A reddish-brown solid (resinous matter) appeared in the solution during the addition of NaBH4. The contents were cooled and isopropanol-soluble fraction was removed and cooled to room temperature. Light yellow crystals of wax were separated from the isopropanol-soluble fraction by filtering. The filtered cake was washed with fresh isopropanol followed by water. After vacuum drying, the cake yielded odor-free light yellow wax crystals.[10]

Gopalkrishna developed a process for extraction in which the step of percolation of solvent through wax suspension was eliminated. The method developed is a combination of physical and chemical methods. Low-pressure and non-polar solvent treatment of wax sludge is carried out to separate wax and oil fractions. The wax fraction thus obtained is of >95% purity. The upgraded wax did not contain phospholipids and had a melting point, hardness, and brittleness comparable to carnauba wax. The wax so prepared in two steps did not require any bleaching. The solvent and oil can be recovered and reused, rendering the process economical and quick. The process includes; hydraulic pressing of wax using a hydraulic press; extraction of deoiled wax-sludge with non-polar solvent in a ratio of about 1:2 under occasional stirring at room temperature for about 30 minutes and collection of wax layer and solvent removal from the said wax layer under vacuum to obtain the purified wax.[11]

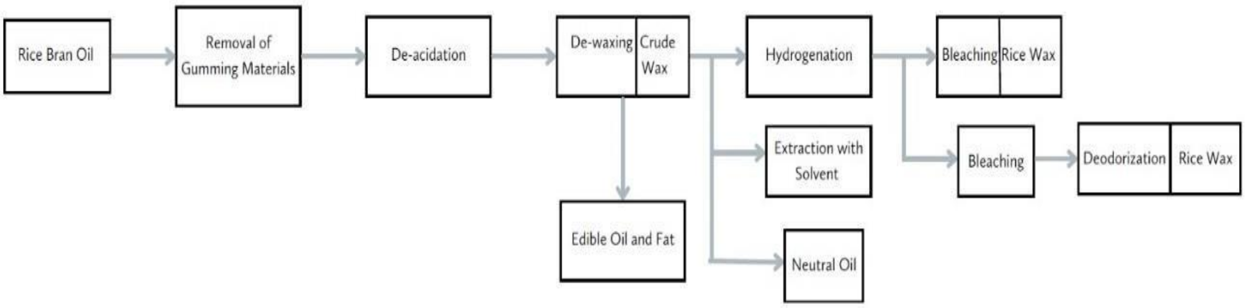
Akira extracted and refined the hard and soft wax from the rice oil by dissolving the crude bran wax oil in isopropyl alcohol and separating the soft and hard wax and gums by the difference in their solubility at different temperatures. Crude bran wax oil was dissolved in IPA and separated at three stages: room temperature (approximately 25℃), 50℃, and 70℃. The room temperature soluble fraction was bran oil and fatty acids, the 50℃ soluble fraction was soft bran wax, the 70℃ soluble fraction was hard bran wax, and the 70℃ insoluble fraction was gum. The obtained bran wax (hard) had a saponification value of 71.3, an iodine value of 9.8, an acid value of 0.7, a melting point of 79.1-80.3°C, and did not contain phosphatides. This method did not require any preliminary treatments such as deoiling with acetone or saponification to decompose and remove phosphatides.[12]

Pominski's invention relates to the production of hard rice wax. Rice bran contains 15 to 20% lipids. The major components of lipids are rice oil, wax, free fatty acids, and phosphatides. The solvent extraction of rice bran removes about 90 - 95% of the lipids. When the oil obtained by such a solvent extraction is freed of oil solvent and allowed to stand, an oil-insoluble tank settling precipitates which amounts to 1 to 2% by weight of the oil and a relatively soft wax fraction, free fatty acids and phosphatides. The solid and liquid portions of such tank settling could not be readily separated by filtration. Dilution with liquid hydrocarbons shows little improvement in their filtration characteristics. However, by purification and fractional crystallization from isopropyl alcohol, a hard rice bran wax, which is comparable to carnauba wax in many of its properties, can be isolated from rice bran oil tank settling. In this process, rice bran was cooked with water to achieve the moisture content of the miscella to about 5% to 7%, which was then treated with an oil-soluble solvent to separate oil and cooled to form an immiscible wax-rich phase, consisting essentially of rice bran wax. This wax was centrifugally separated to recover the wax and solvent. In another method, the residual bran devoid of rice oil, was treated with hexane. The miscella was then cooled to form the immiscible wax-rich phase. The wax and solvent were recovered via centrifugation. The resultant wax was repeatedly washed with hexane to remove traces of hexane-soluble components from the wax- rich phase. Separation can also be achieved mechanically either by decantation or centrifugation, which was aided by the difference in their specific gravity though preferred method of separation is centrifugation.[13]

Buffa collected liquid oil and fat and solid oil and fat separately from the unhulled rice and used two different processes to refine them. In the first process, he extracted the solid oil and fat portion with solvent. The wax was then bleached and refined without deterioration of its inherent characteristics. In another process, the resultant wax after bleaching was subsequently deodorized. The resultant wax from both processes varied in their physical properties and chemical composition.[14]

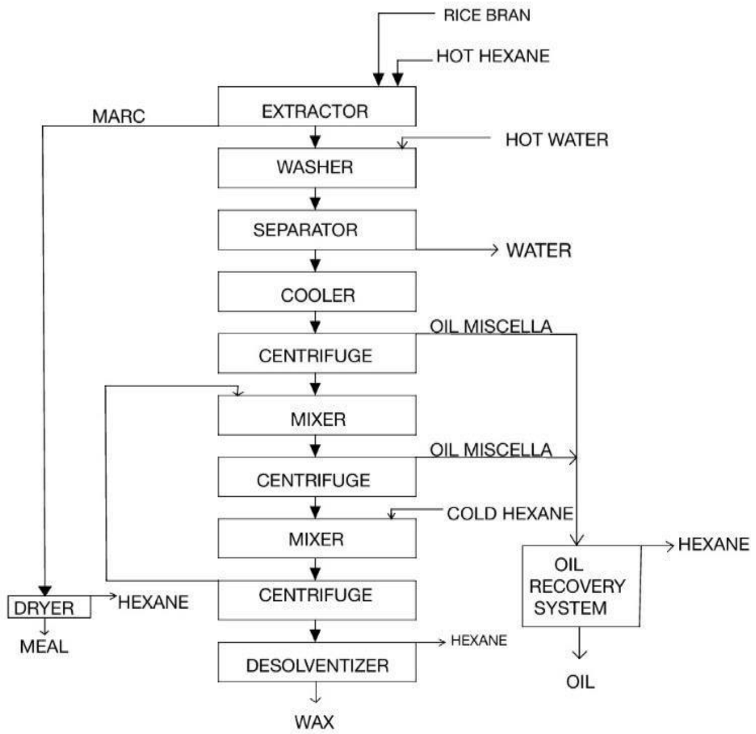



**Flow Chart 1: Refining system by Charles Buffa type 1.[14]**

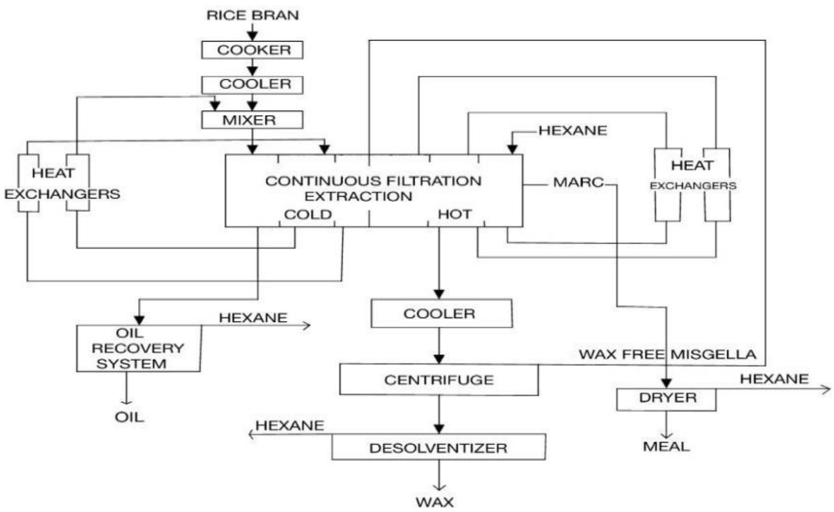



**Flow Chart 1: Refining system by Charles Buffa type 2.[14]**

Pominski in another study explained two different methods for simultaneous recovery of wax and oil bran by filtration-extraction from rice. In the first method, preferential cold hexane extraction was carried out to remove oil from the bran, followed by hot extraction to remove wax. The wax miscella was then cooled and the precipitated wax was recovered by centrifugation. In the second method, a single hot solvent extraction of rice bran was employed to remove wax and oil simultaneously. Wax was separated from the oil and wax miscella either directly or after a hot water washing of the miscella, by chilling, settling or centrifuging, and desolventizing. If rice bran is cooked, both hot and cold solvent extractions can be performed in a single extractor of the filtration-extraction since the filtration rates are high and within practical limits. This First method has the advantage that no water or solvent-washing is necessary to produce purified wax, and probably only one centrifuge would be required. In the second method, multiple cold solvent washes would need to be carried out after hot water washing to purify the wax, which necessitates the use of several centrifuges.[15]




**Flow Chart 3: Flow diagram for simultaneous production of rice wax and oil by a single hot extraction.[15]**




**Flow Chart 4: Flow diagram for filtration extraction process for simultaneous production of rice wax and oil.[15]**

Green Spring uses a three-step process for rice bran wax refining viz; degumming, degreasing, and decolorization. Degumming is achieved by reacting the molten wax with phosphoric acid and water vapor. The reaction was allowed for a certain period by keeping the solution warm and static. The lower layer of precipitated gelatin is removed. After degumming, the content of gum and other impurities were found to be less than 0.05%. The organic solvent is added to the degummed crude wax at a set temperature and cooled gradually. The constant temperature is maintained for 2 hrs at each required cooling temperature, the precipitate is collected at each temperature point and the sample is analyzed for purity. The degummed and degreased wax obtained is brown in color. The decolorization is done in 2 steps. In the first step the wax is bleached with hydrogen peroxide followed by further bleaching with sodium hypochlorite. The wax is then washed to neutral, and its whiteness is determined. Phosphoric acid hydration and degumming, ethyl acetate extraction and oil removal, and two-step decolorization treatment, provided a better raw material for the further use of refined rice bran wax.[8]

Rice bran wax can also be refined through batch chromatography technology as this retains policosanols, phospholipids, phytosterols, and squalene which are present in very low concentrations in the wax. The resulting rice bran wax is of superior quality.[16]

Pemac projects use mechanical and solvent extraction for refining crude wax. The former involves a top-loading vertical plate type press machine operated with the help of hydraulics and has the advantage of low electricity consumption, lower maintenance cost, quick filtering cycle, and ease of use, but has the disadvantage of low recovery of oil from wax and low- quality wax production which can be overcome by the solvent extraction, followed by the mechanical extraction method. The solvent extraction involves multiple stages of extraction to remove residual oil content of up to 98%, lowering the iodine value and unsaturated lipid content, the free fatty acid content of the wax composites, and extending their shelf life. In the final step, the de-oiled and defatted wax composite was extracted again with solvent to precipitate the residual matter. Filtration was carried out to remove residual leftovers, the solvent was eliminated by drying it. The resultant wax is light yellow with low density. The whole process is carried out in a controlled environment to achieve less than 4% oil in wax and remove all the impurities.[17]

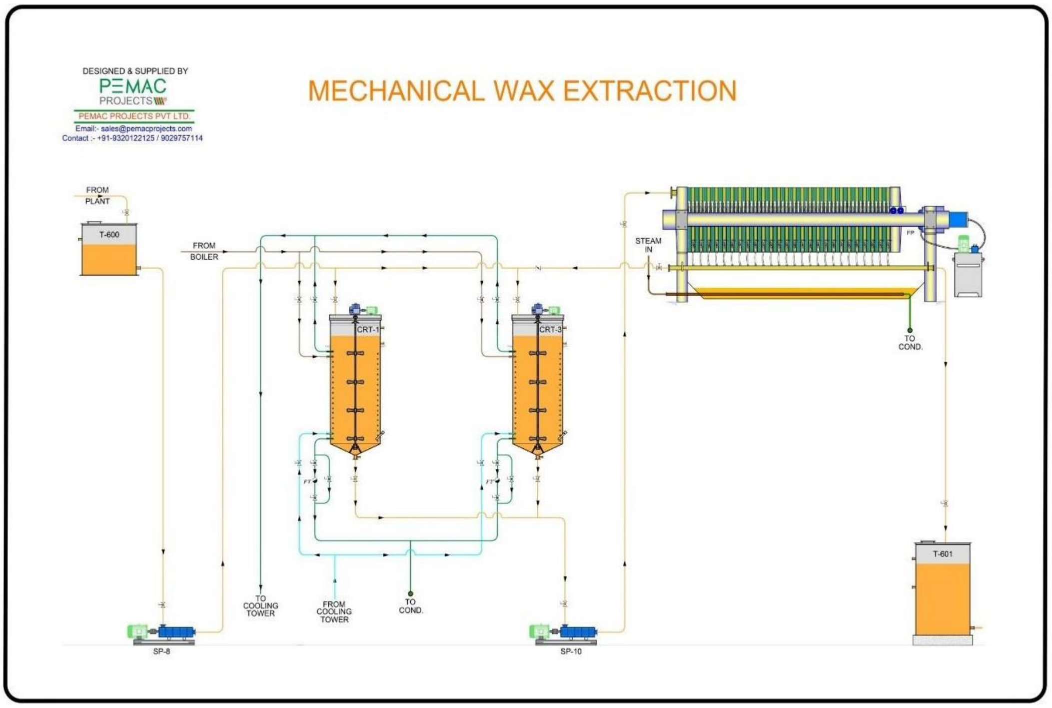



**Fig. 1: Wax extraction plant, Pemac projects.**

**DISCUSSION**

The percentage range of lipids in rice bran is about 14–17% oil of which 3-9% is wax.[15] The rice bran lipid consists of rice oil, wax, free fatty acids, and phosphatides. The solvent extraction of rice bran removes about 90 to 95% of the lipids. When the oil obtained by such a solvent extraction is freed of oil solvent and allowed to stand, an oil-insoluble tank settling precipitates which amounts to about 1 to 2% by weight of the oil and consists essentially of oil, a relatively soft wax fraction, free fatty acids and phosphatide along with meal, trash, and calcium carbonate which is used in milling of rice, gums, resins, and degraded organic matter in minor amounts. The solid and liquid portions of such tank settling cannot readily be separated by filtration. Dilution with liquid hydrocarbons such as hexane, heptane, and the like, provides little improvement in their filtration characteristics. The crude wax obtained during the refining still contains a good percentage of oil, phosphatides, resinous matter, and other impurities.

Different methods have been developed for refining the crude wax like solvent extraction, cold and hot extraction, batch chromatography, mechanical extraction, fractionation, etc. As mentioned in Bailley's, a simple extraction at varying temperatures can help separate the soft and hard wax and the gum followed by filtration and centrifugation. The resultant wax will further need deodorization and bleaching for further refining.[9] Vali proposed a two-step process for refining viz; defatting of crude rice bran wax and bleaching of the defatted wax. Hexane, isopropanol, and sodium borohydride ($NaBH_4$) solution in isopropanol were used and an odor-free pale-yellow wax was obtained.[10] Gopalakrishna developed a combination of physical and chemical methods to obtain more than 95% pure wax.[11] Akira used the solubility characteristics at different temperatures to produce soft and hard wax and remove gums. The proposed method did not require preliminary treatments such as deoiling with acetone or saponification to decompose and remove phosphatides.[12] Pominski used solvent extraction and centrifugation/decantation to obtain the purified wax. He relied on the difference in specific gravity of the components for their separation.[13] Buffa used two different purification methods with the difference of an additional step of deodorization on liquid oil and fat and solid oil and fat components.[14] Pominski in another paper proposed the use of selective cold and hot hexane extraction method and a single hot extraction method wherein he extracted oil and wax simultaneously. The selective hot and cold extraction process was preferred as it has the advantage of the use of a single extractor for the filtration-extraction, higher filtration rates within practical limits and no water or solvent washings were necessary for further purification of the wax.[15] Green Spring proposed a three-step process for rice bran wax refining viz; degumming, degreasing, and decolorization for the refining of crude wax.[8] Pemac projects uses mechanical and solvent extraction for the refining of crude wax. They emphasize that to get a purer grade, the combination of physical and chemical methods is necessary.[16]

As can be seen, almost all scientists use solvent extraction methods to purify the crude wax with some relying on a combination of mechanical and chemical methods while others used only chemical process for wax purification. The solvents used are normally selected based on solubility. It is observed that higher solubility of wax in a solvent was a hindrance for some while others used that characteristic to their advantage. The use of activated clay or carbon had little effect on the color improvement and removal of resinous matter and thus chemical bleaching became a necessity. Different scientists have used either strong or mild bleaching agents. The disadvantage of using strong bleaching agents is that the process if not controlled can be very violent and may transform the wax chemically while the probability of the same is very minute when using mild bleaching agents. Some scientists have developed processes where the waste generation is minimal and the by-products of wax refining can be recycled. The oil recovered during the refining process can be added back to the oil, solvents can be recovered and reused for further batch extractions, and oxidizing agents recovered by electrolysis during the refining process can be reused. These processes are sustainable and eco-friendly.

## CONCLUSION

After reviewing all the outlined and proposed methods, it can be safely concluded that the best method for the extraction and refining of rice bran wax is a combination of mechanical and chemical processes. Fewer mechanical processes may reduce raw material loss and lower chemical processing may help in reducing the processing time and cost. One may choose a process that can also help recover the oil present in the wax without alternating its chemical structure and composition so that it can be added back for refining with the oil. Solvent selection, temperature, cost of chemicals, and reaction time can be big determinant factors in chemical processing as all of these may adversely affect the final product. One may also consider a process that can help in recovering chemicals used which can be recycled and reused. The chemicals used should not lead to violent reactions as they may prove to be dangerous if not controlled and may transform the wax chemically.

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