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A GREEN TECHNIQUE FOR NANO-CELLULOSE DERIVATIVES PREPARATION AND FUNCTIONALITY

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ABSTRACT

Using a ball mill is a low-cost, easy, and fast green solution that has a lot of promise for expansion. The manufacture and chemical manipulation of microcrystalline cellulose and nanofibers is amongst the most intriguing uses of these approaches in the area of cellulose. Ball milling is a method of reducing nanotubes to ultrafine particles by grinding them. While the ball milling process is in progress, the collision of small hard balls in a concealed jar results in the generation of localized high pressure. Ceramic, flint stones, and stainless steel are all often utilized in this process. When it comes to grinding (or mixing) materials, ball mills are cylindrical in form and are used for a variety of applications such as ore grinding (or mixing), chemicals grinding (or mixing), ceramic raw materials grinding (or mixing), and paint grinding. Ball mills are cylindrical devices that spin around a horizontal axis and are partially loaded with the material to be treated as well as with the grinding media. Despite the publication of a number of papers, the potential of this method in the area of cellulose nanoparticles has not yet been fully realized. Aiming to put current work into perspective, this analytical study emphasizes the significance and potential of this renewable, renewable approach for identifying areas for future development, as well as the challenges and opportunities it presents.

KEYWORDS: *Amorphous, Ball milling, Cellulose, Nanocrystals, Nanocomposites.*

1. INTRODUCTION

An increasing understanding of the importance of ecologically sustainable chemical product and process design has emerged in recent years. The concept of sustainability is having a significant impact on the chemical industry, which is gradually shifting its focus away from the use of toxic chemicals and toward the incorporation of green synthetic solutions that start with natural, organic resources as their starting materials. A great deal of interest has been generated in this regard by the fact that cellulose is a naturally occurring, abundant, cost-effective, biocompatible, and biodegradable natural fiber that has a wide range of applications in a variety of fields such as papermaking, textiles, coatings, pharmaceuticals, implant manufacturing, and tissue engineering.

This linear homopolysaccharide is made up of glucose units connected together by (1-4)-glycosidic bonding. Hydrogen bonds as well as Vander-waals Forces between cellulose chains produce elementary fibrils, which subsequently aggregate into bigger micro- as well as nanofibrils 2–10 nm in diameter, respectively. It is possible for the cellulose chains included inside these aggregates to be organized in either chaotic amorphous-like areas or highly structured crystalline structures. A variety of polymorphs of cellulose can be formed depending on the dimensions of the unit cell as well as the chain polarity of the chain(1). Native cellulose contains cellulose I, whereas cellulose II crystallizes after many treatment steps. Cellulose nanofibres (CNF) aggregation that comprise both amorphous and crystallized regions are known as micro fibrillated cellulose (MFC). MFC may be manufactured from raw materials using a mix of chemical and mechanical techniques. Nanofibres may be formed via mechanical shearing operations such as elevated homogenization, strong and effective, mini, high-intensity ultrasonic therapies, and grinding. As an aid in the removal of lignin and hemicellulose from wood, chemical therapies (such as acid hydrolysis, TPO-mediated oxidation, and enzymatic reactions) as well as biological pretreatments are available. Concentrations as low as 1 percent weight can result in extremely viscous, entangled networks of fibrils in aqueous media, and these networks can be extremely difficult to break up. Different types of bacteria have the ability to produce extremely crystalline cellulose nanofibres as well (e.g. Komagataeibacter Xylinus). In this case, the fibers are excreted at the air-water interface after the plants have been grown in aqueous nutrient media. In addition to having a high weight-average molecular weight, Bacterial Nano Celluloses (BNCs) are also mechanically stable(2).

The extraction of crystalline areas present within cellulose micro-fibrils enables for the gathering of crystalline rod-like particles known as cellulose nanocrystals, which are crystalline rod-like particles with a crystalline rod-like structure (CNCs). Acid hydrolysis, which eliminates amorphous regions and breaks down hydrogen bonds, is indeed the primary method by that they are made, among other things. CNCs have a lower aspect ratio than cellulose nanofibers (CNFs) and are more crystalline than these fibers. All three types of CNFs, CNCs, and BNCs are clubbed together under the name "Nano celluloses," which is used to refer to cellulosic extracts or processed products with a single dimension measured on the nanometer scale. When compared to the other mechano-chemical processing techniques available for cellulose, ball milling is a newer approach that avoids the use of organic solvents and allows for the reduction of cellulose fiber size. In recent years, this method has acquired growing appeal in the field of chemistry because to its simplicity of use, rapidity, economy, and environmentally friendly nature. Ball milling is used in the processing and chemical functionalization of cellulose nanofibres and nanocrystals, and this article will discuss some of the newer advancements in this field. Research on the application of ball milling in manufacturing engineer, specialty chemicals, and biodegradable and polymer nanocomposites has already been published, among other areas of study. The technique's promise in the field of cellulose nanoparticles, on the other hand, has not yet been thoroughly explored. By emphasizing the significance and promise of this renewable, sustainable approach for identifying areas for future development, the purpose of this paper is to put present efforts into sharper focus and to bring it into the public eye(3).

Ball milling is a low-cost, simple, and quick green technology that has great potential for growth. The research that were reviewed showed that it can be used in the production and chemical functionalization of cellulose nanocrystals and nanofibres. Another important advantage of this method is its ability to be combined with chemical treatments, which allows for the production of desired products with little effort. However, there are a number of considerations that must be taken into account in order to determine how the characteristics of the isolated Nano celluloses will be affected. It is thus necessary to optimize the process in order to distinguish CNCs with the required characteristics from the others. It is possible to affect the crystallinity of isolated nanoparticles, the rate of thermal degradation, the volume, the thickness, and the shape of the nanoparticles in isolation. If the provenance of the raw material isn't taken into account, the process's efficacy and features of Nano cellulose obtained may be significantly affected, thus it is important to choose the source of the raw starting material carefully(4).

1.1 Fundamentals of A Ball Mill:

As well as being known as a ball mill, a pebble mill, or tumbling mill, a ball mill is an industrial milling device that is comprised of a hollow cylindrical shell filled with balls, which is attached to a steel frame that may be spun along its longitudinal axis. It is estimated that the spheres, which may have varying diameters, take up 30 – 50% of the mill's volume, with their size determined by the feed and mill size. Large balls have a tendency to break down coarse feed products, while tiny balls assist in the production of fine material by decreasing the amount of free space between each ball. Ball mills grind material by using the forces of impact and attrition. The following variables have an impact on the amount of milling that occurs in a ball mill:

1) The amount of time that the material spends in the mill chamber.
2) The number of spheres, their height, and their density are all different.
3) The balls are made up of a variety of materials (hardness of the grinding material).
4) Feed rate and vessel feed level are also important considerations.
5) The rotating speed of the cylinder.

There are many distinct kinds of ball mills, and the operating principle of each varies to a certain extent. Aspects of their design differ as well, such as the size of the total milling vessel, which may range from less than a liter for planetary ball mills, mixer ball mills, and momentum ball mills to several hundred liters for horizontal rolling ball mills(5).

1.2 Applications of Ball Mill in Pharmaceuticals:

1) Small to medium-sized ball mills may be used for final grinding of medicines, or even for grinding suspensions, and can be found in a variety of configurations.
2) Ball mills with the greatest possible capacity are used to process ores prior to the production of pharmaceutical compounds.
3) Ball mills provide a number of advantages.
4) It produces a fine powder as a result (particle size less than or equal to 10 microns).
5) It has the capability of being utilized completely sealed, making it suitable for grinding hazardous materials.
6) It may be used to a number of different purposes.
7) It has the capability of providing continuous service.
8) It is used in the grinding of very abrasive materials.
9) Ball mills have a number of disadvantages.
10) Damage to the material may develop as a consequence of normal wear and tear, which is mainly caused by the balls and to a lesser extent by the packing.
11) Especially when the hollow cylinder is constructed of metal, machine noise is significant; however, when rubber is utilised, machine noise is much reduced.
12) The milling process takes an unusually lengthy period.
13) After a computer has been used, it is difficult to clean it completely.

2. DISCUSSION

A ball mill is indeed a common mechanical method for finely grinding and combining powders and other materials. It is widely used in industries across the world since it is both environmentally benign and cost-effective. A ball milling procedure for processing and functionalizing Nano cellulose derivatives has been examined in this research instead of the equipment themselves, it will not go into detail about the different kinds of machines available in the industrial setting. In spite of it, this part offers a high-level introduction to the many kinds of machinery. Ball mills are available in a variety of configurations to suit a variety of applications. In most cases, however, it is comprised of a hollow cylindrical shell that spins around its axis and is partially filled with balls composed of steel, stainless steel, ceramic, or rubber, with the remaining space filled with air. A ball mill (grinding or milling medium) and a powder are used to generate energy via the action and attrition of the balls (grinding medium). Among the advantages of this technique are its cost-effectiveness, efficiency, simplicity of operation, repeatable results owing to energy and speed control, and application in both wet and dry conditions on a broad range of materials, among others (e.g. cellulose, chemicals, fibers, polymers, hydroxyapatite, metal oxides, pigments, catalysts). On the other hand, potential disadvantages include the possibility of corrosion, the formation of nanomaterials with odd shapes, noise, and the need for lengthy cleaning and milling periods(6).

Depending on what kind of material to be produced, there are a variety of different types of machinery available. Direct milling as well as oblique milling are now the two types of ball mills that may be used. To transmit kinetic energy towards the particles in the first scenario, rollers or motorized shafts apply direct force on them. The energy released is first transmitted to the mill body, and from there to the grinding medium. The three most prevalent kinds of ball mills inside the cellulose sector are tumbler mills, vibrating mills, and cosmic mills. It is the most frequent form of planetary ball used for the cellulose business, and tumbler powder metallurgy, vibrating mills, and planet mills are further subdivided into three categories (7). There is a barrel with steel balls within that rotates itself along the long direction in a tumbler mill, with the steel balls acting as grinding media. The diameter of the mill has a significant role in determining the efficiency of the operation in this kind of instrument. As a result, greater diameters help in increasing fall heights, resulting in more impulse being delivered to the balls. The tank carrying the sample or the ground medium is rocked back and forth through high energy bands during the working of vibratory mills. This scenario necessitates the study of many factors, including the frequency of vibration, its amplitude, and the mass of the milling medium. To finish it off, the containers are each supported by a spinning disc and each rotates around its own axis in a planetary mill. It is important to note once again that the size of the vessels is a key factor in the process's effectiveness since a wider vessel width allows for greater kinetic energy and therefore, more powerful impacts(8).

One of the most fascinating uses of something like the ball mill inside the field of cellulose is the production of CNCs and CNFs. This technique offers a number of benefits over other methods for producing Nano celluloses and cellulose nanocomposites, including the fact that it is cheap, easy to use, and quick. Although the working parameters must be sensiblyscrutinized to evade over-grinding of the raw materials, this is not always the case with commercial equipment. When used properly and in conjunction with chemical and enzymatic treatment, this approach may also be used to induce cellulose degradation and de-polymerization. To make cellulose nanocrystals, it is necessary to hydrolyze native cellulose using mineral acids, which destroys the amorphous cellulose and liberates the crystalline portions. This process is aided by shearing actions. Ball mills are excellent tools for integrating mechanical or chemical behaviour in the production of CNCs because they apply mechanical forces while also containing chemical agents. All of the variables, including the acid used for hydrolysis, the pre-treatments employed, and the duration and speed of milling, should be addressed since they may all have an impact on the crystallinity, shape, and size of the CNCs that are extracted(9).

After submitting the cellulose to a mechano-chemical procedure in the presence of acid, the researchers used a ball mill to extract nanocrystals. To eliminate any leftover fibers, the pulp was first pre-treated using a Fiber Dissociation Unit. After collecting the material, ball milling it at 200c for 1.5–3.5 hr in the condition of phosphoric acid produced a clear solution. Phosphoric acid was utilized as an enlarging agent during cellulose dissolving process in this mechano-chemical approach, enabling for easier breakdown of cellulosic fibers into nanofibers. Following that, quadruple water was added to isolate the fiber from the previously developed gelatinous precipitation. A set of centrifugations were performed by an ultra-sonication procedure at 50 °C for 1–3 hours to remove cellulose nanocrystals. The particles formed were revealed to be mostly composed of the viscose type-II polymorph, with length and width ranging from 100–200 nm as well as 13–30 nm, respectively. As a consequence of their prior work, the same study team subsequently devised a technique for extracting nanoparticles from bamboo pulp by mechano-chemical reaction in the context of plasmon acid.

After milling for 1.5–2.5 hours, both purification and ultra-sonication processes were completed. With their usual rod-like structure and curled flat curvature, the resulting nanocrystals averaged 200–300 nm in lengths and 25–50 nm in breadth. The concentration of phosphorus-tungstic acid, the reaction duration, and the milling period were all critical factors in the process. Longer reaction durations at a constant acid concentration resulted in higher yields of isolated CNCs, following 4.5–5 hours under optimum circumstances, optimal conditions are reached. The yield was also increased by increasing the phosphor-tungstic acid content in the solutions from 10% to 15%. Because of this, it was simpler to hydrolyze the amorphous portions of the cellulose and remove the nanocrystals from the sample. The yield of CNCs dropped when the acid concentration was increased, in part due to the complex formation of the ball milling operation and the acidity in cellulose hydrolysis inside the vicinity. When used in conjunction with chemical agents, mechanical ripping may be performed without the need of specialized equipment, as previously mentioned. Thus, this technique may be utilized not only to extract cellulose nanocrystals but also to functionalize them as a consequence of its versatility. However, it is critical to optimize the technique that is being used since the milling length and rotation speed have an impact on Isolated-crystal production yield and substitution degree.When working with CNCs in the presence of maleic anhydride, for example, researchers discovered a solvent-free technique for extracting and chemically altering CNCs. We utilized this technique for the processing of filter paper cellulose pulp, which was mechano-chemically treated at 500 rpm for 0.5–2 hours in a planetary ball mill with maleic anhydride and sulphuric acid for 0.5–2 hours. In order to improve the performance of the chemical reaction, an ultrasonic reactor technique was used in conjunction with this. Both the yield (the proportion of nanocrystals generated from cellulose pulp) and the degree of substitution of the CNCs produced by the ball mill process were affected by the ball mill technique. Although only 16.3 percent yield was obtained when no ball milling was used, the yield increased to 56.1–61.1 percent after ball milling for 0.5–1 hour(10).

This increase is most likely due to the breakage of inter- and intramolecular hydrogen bonds, which results in an increased amount of hydroxyl groups required to react. A 2-hour reaction time resulted in needless degradation of the cellulose crystalline structure, and the yield was reduced by 54.7 percent, according to the results of the study. Similarly, when the milling time was extended to one hour, the degree of substitution values altered, and when the operation duration was prolonged to two hours, the degree of substitution values dropped. Prior to functionalization, esterification of the crystal's most accessible areas happened first, followed by esterification of the crystal's less accessible regions. Variations in crystallinity were also found to exist. The crystallinity increased as a result of the disintegration of cellulose's porous structure during the reactions. There are two types of polymer nanocomposites: those that have a nanoscale component and those that have a microscopic component. They may be used to improve or customize certain features of mixture components, thereby broadening the spectrum of what could be accomplished with them and overall range of possibilities. Because of their broad dispersion, varied characteristics, and cheap cost, Nano-celluloses are attractive candidates for use as fillers in nanocomposites when coupled with other polymers. Nano-celluloses are particularly attractive as fillers in nanocomposites when combined with other polymers. When designing such hybrid systems, the ball mill is a straightforward tool since it allows for the utilization of mechanical shearing behaviour in the presence of chemical agents. A particular instance was documented, in which the researchers developed a polymer nanocomposite made of cellulose nanocrystals and ultrahigh molecular weight polyethylene (UHMWPE). It took six hours at 200 rpm rotating speed in a planetary ball mill to generate this nanocomposite, for which commercially available CNCs and UHMWPE were combined with six hours of grinding to create. Disk or dumbbell-shaped derivatives with increased biocompatibility and the potential to be employed as bearing components for knee replacements were hot-pressed from the resulting mixture of components.

3. CONCLUSION

There has been a dearth of research on the possibilities of ball milling techniques to separate and chemically alter cellulose nanoparticles. Process engineering, chemical synthesis, and nanocomposites have all benefitted from ball milling technology. Because of their poor solubility in organic solvents, further study into the functionalization of CNCs would be especially intriguing, since these materials are very challenging to react with because of their low solubility. Aside from esterification, various chemical transformations may be researched and reported; however, these investigations are likely to be limited by the presence of functional groups in solution. There may be more processes in which the cellulose hydroxyl groups serve as a nucleophile in addition to the ones described above. Ball milling, in addition, since it can be used in both dry and wet settings, has the potential to be a renewable and environmentally friendly industrial technology in the future. Research into cellulose-based nanocomposites is also of interest since this method provides a straightforward and rapid approach to generate such hybrid systems, which is very important. As an important technology, it is expected that this research will help to recognize and identify areas for future development its relevance and potential.

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