

MECHANOCHEMICAL PRODUCTION OF LIGNIN-CONTAINING POWDER FUELS FROM BIOTECHNOLOGY INDUSTRY WASTE A review

by

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In biotechnological processing of plant raw materials, carbohydrates that are soluble and accessible for microorganisms are the only usable components. The lignin-rich part of the plant raw materials usually ends up in the waste. Lignin transferred into water suspensions cannot be used efficiently as a fuel. In this review, a new processing scheme of plant raw materials is presented, which includes mechanochemical treatment of the plant raw materials and separation of the powder product into particles of lignified and non-lignified tissues rich in lignin and cellulose, respectively. The cellulose-rich powders can then be used in biotechnological processes. Lignin-rich powder aerodynamically separated using cyclone-type apparatus can be used as a powder fuel to satisfy the needs of the main biotechnological plant in heat and steam.

Key words: *lignocellulosic fuel, mechanochemistry, grinding, lignin burning, agricultural wastes, biotechnological industry*

Introduction

The importance of developing processing technologies of lignin-containing waste is clearly visible from the fact that per each kilogram of grain product on average produced by the agricultural industry 5 kg of lignocellulose waste form, which are used inefficiently or burnt in the fields.

The potential feeding value of the products based on carbohydrates remaining in this amount of waste can be 2-3 times greater than that of the grain products. However, carbohydrates contained in lignocellulosic biomass are insoluble and cannot be hydrolyzed; for this reason they are indigestible for most animals and microorganisms. Cellulose-based components separated from the plant raw materials can be used as high-quality feed in husbandry and as a feed source for the growth of microorganisms. They can also serve as a biorenewable basis for the chemical industry. In the USA, according to "Green Chemistry" program, 25% of the chemical industry is planned to be turned to biorenewable plant raw materials. A problem is the high

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cost for realization of existing technologies. The decreasing of treatment cost especially as a result of non-cellulose components is a possible solution.

Lignin, a component of lignocellulose composite, is of polyphenol nature and is a ballast material from a food chemistry viewpoint. As the calorific capacity of lignin is higher than that of carbohydrates and cellulose, it could be efficiently used in power engineering. However, the currently existing lignin separation technology from plant raw materials is based on sulphite pulping and is associated with high levels of sulfur contamination greatly exceeding the values allowed for the fuels.

Advanced processing technologies of plant raw materials and wood should include:

- production of substances containing biodigestible carbohydrates,
- utilization of lignin waste, and
- energy supply for the primary technology of carbohydrates.

The main aim of this work was to demonstrate the potential ability for separate plant material into fractions and further burning lignified fraction as a solid powder fuel.

Processing technologies of lignocellulosic materials and related equipment

Several processing methods of plant raw materials have been developed to obtain soluble carbohydrates [1, 2]:

- dissolution of lignin by chemical reagents such as caustic alkalis, ammonia, chlorites, sulfur dioxide, amides, dilute and concentrated acids, which is widely used in the production of pulp and paper [3],
- explosive autohydrolysis [2, 4],
- enzyme treatment or treatment by enzyme-producing fungus strains [5],
- microwave radiation [6],
- ultrasound treatment [7],
- electron beam treatment [8], and
- γ -radiation [9].

Methods associated with radiation are not widely used because of their prohibitive cost.

Technologies of feed production from straw use mechanical treatment [10, 11]. Straw and other plant raw materials are cut with knives and then gripped by blades fixed at the opposite sides of the rotor to be pushed to the next set of knives. Unfortunately, the cutting devices cannot produce particles smaller than 200 μm . Nor can they destroy the structure of the plant cells having lignocelluloses walls 5 μm thick.

Grinding mills with a wide distribution of milling bodies over the intensity of impact (ball mills, attritors) have limited application in the treatment of plant raw materials. High-energy impacts [12] lead to non-selective destruction of plant tissues. The requirements to the intensity of impact, the dwell time of the raw materials in the treatment zone and the temperature of the chamber of the milling device are satisfied in centrifugal roller mills [13]. Treatment in this mill results in the destruction of plant raw materials into particles ranging from 10 to 40 μm . Each particle is a composite particle and consists of the fragments of the lignocellulosic cell walls 50-200 nm in size [14].

The composite particles formed during milling of the plant tissues are either rich in fragments of the cellulose cells, fig. 1(a), or rich in fragments of the lignified cells containing lignin, fig. 1(b) [14]. Particles differing in cellulose and lignin contents differ in specific weight. The powders produced by grinding in a centrifugal roller mill can be separated into fractions of

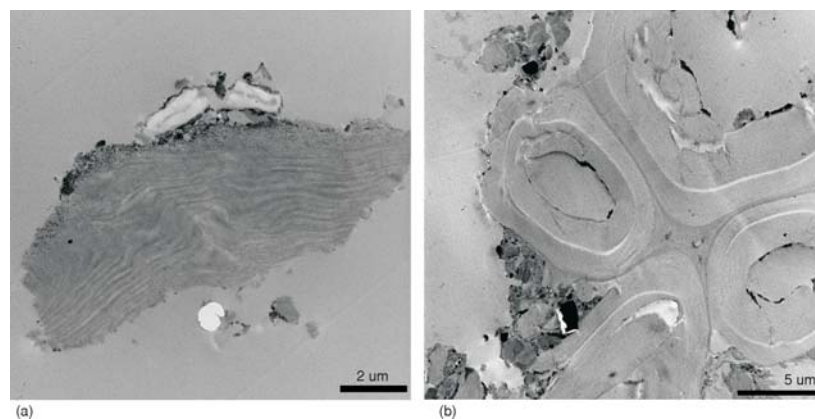


Figure 1. The structure of a cellulose particle from a low-lignified plant tissue (a) and the structure of a high-lignified particle from a lignified tissue (b)

different density and thus enriched in either lignin or cellulose using cyclone separation set-ups [15]. It is particularly noteworthy that the aerodynamic separation is a completely new method. At this moment only Jackering company” succeeded in obtaining starch from the grain into an industrial scale [16].

Utilization of lignin contained in plant raw materials as an energy supply in the biotechnological processing of lignocellulose

Mechanochemical treatment of lignocellulose raw materials and separation of the ground product into cellulose-rich and lignin-rich fractions allows increasing the processing efficiency of plant materials by reclaiming the lignin waste and utilizing it as fuel in the same biotechnology industry.

Several methods of concentration of plant raw materials are known, some of them are based on the dissolution of lignin and its removal, analogously to the process of paper production. Such methods contaminate lignin with sulfur- and chlorine-containing compounds, which makes it unsuitable for the energy applications. Burning of these compounds would lead to highly toxic emissions into the atmosphere.

Alternative processing methods are currently being developed using solution-based technologies and solid-state enzymatic treatment, which exclude contamination of lignin with sulfur or chlorine. Methods of concentration of the plant raw materials are also being developed using separation of plant tissues differing in chemical composition.

The main physical and chemical difficulties of the production of fuels from non-edible biorenewable raw materials stem from the reasons discussed below. In plant raw materials, lignin is non-uniformly distributed over different plant tissues. Non-lignified tissues contain negligible amounts of lignin, but the technology of their separation from the lignified tissues has been developed only for certain types of plant raw materials on a laboratory scale. However, this presents a possibility of using solid-state processes to grind the plant raw materials into particles differing in chemical composition and subject them to aerodynamic separation.

In lignified tissues, lignin is located in the intercellular space and is bound in complexes with polysaccharides. In order to separate compounds bound at the molecular level, liquid phase processes are used, which are costly and cause ecological problems due to large amounts of waste water.

Burning is currently the main utilization method of wood waste. Although a certain amount of biomass is used for heating and cooking purposes, the production of energy using burning of biomass is much less developed [17]. The disadvantages of the fuel produced from biomass such as:

- instability of composition and calorific capacity,
- difficulties of controlling the burning rates,
- difficulties of supplying the fuel at a constant flow, and
- high investments needed for boiler set-ups.

Limit the wide use of biomass for the energy production. Some of these disadvantages are related to the heterogeneity and low density of the plant raw materials. In the recent years, attempts have been made to increase the homogeneity of the biomass-based fuel by making pellets. To produce pellets, biomass is ground, then different components are mixed in the powder state and pressed into pellets suitable to be poured and fed into the reactor [18, 19].

Using pellets and briquettes of biomass in modernized grate furnaces has improved the burning parameters (efficiency, temperature, stability) and reduced the carry-over of unburnt fuel. Fuel transportation and storage as well as feed of the furnaces have become easier. However, the biomass briquettes are lump fuel and show low evolution rates of volatile components and heat [20, 21]. A solution to this problem would be a powder fuel made of biomass.

One of the main challenges of using the powder fuels is to prevent agglomeration of the powder particles [22]. Particles smaller than 1 μm are prone to agglomeration. Particles that have not been burnt completely deposit on the equipment surfaces forming a slag negatively influencing the process.

In order to separate a powder fuel into fractions with more narrow size distribution, aerodynamic separation can be used. Parameters that determine the density and rheology of a dispersed medium are the particle size, particle sphericity and interparticle interactions in the air flow. Powder particles can be separated from each other by forces exceeding those of the interparticle interaction. However, at present, the nature of forces responsible for agglomeration is not yet fully understood making it difficult to control the dispersion process [23].

Additional difficulties stem from specific features of biomass, such as heterogeneous composition and fibrous structure [24, 25]. Despite all these difficulties, particles of the ground biomass have a particle size distribution and particle shape suitable for performing stable burning [23, 26, 27].

Burning of lignocellulosic powder

The burning process of biomass particles includes following stages [22]: heating, drying, pyrolysis and evolution of the volatile components, combustion of the volatile components, and combustion of carbon.

Each stage has its own time parameters, which greatly depend on external conditions and fuel properties. According to contemporary theories [28], burning of particles includes several processes:

- reactions of solid fuel on the particle surface and in the particle interior,
- secondary reaction of liquid and solid phases on the particle surface and in the particle interior,
- gas-phase reactions on the particle surface and in the particle interior,
- gas and liquid phase diffusion (diffusion in pores) and mixing in the particle,
- mass exchange with the surroundings, and
- heat exchange with the surroundings.

The duration of each stage greatly depends on the water content in the particles, the nature and content of the volatile components and the properties of coal formed after the volatile components have been removed. Different stages are related to each other and can proceed simultaneously under certain external conditions and at certain fuel properties.

After water has been removed at temperatures below 500 K, the volatile components begin to evolve and the pyrolysis stage continues up to 1300 K. During this stage, the particles lose heavy organic compounds. A solid carbon residue forms simultaneously. Volatile components are very reactive and if their concentration is high enough and external conditions favor ignition, flame forms. Burning of volatile components proceeds at a very high rate such that the typical temperature of the particles during this stage is 1500 K.

When burning of biomass particles is considered, the main parameters to take into account are the size of the particles and thermal conductivity and heat capacity, which determine the heat transfer to the particle center, drying processes and evolution of the volatile components. The specific surface is also an important factor as it influences the evolution rate of the volatile components and the burning rate of carbon.

Carbon from biomass is considered to be more reactive than carbon obtained from coal due to a higher specific surface and the presence of radicals on the particle surface [29].

Biomass as a fuel differs from coal by the ash content and composition and its ability to melt. Compounds containing alkali metals, which are responsible for the formation of ash deposits on the surfaces of heat exchangers, are present in biomass in higher contents and can be easily carried over to the gas phase during burning. In biomass, these compounds are salts or they can be bound to organic components. Melting of the ash depends on the presence of alkali metals, phosphorus, chlorine, silicon, and calcium [30]. The formation of ash deposits on the surfaces of heat exchangers is influenced by the following factors:

- fuel composition,
- local gas temperature,
- local heat flow on the particle surface,
- temperature of the heat exchanger tube,
- temperature difference,
- gas velocity, and
- tube orientation.

The growth rate of the deposit layer can be controlled by influencing the reactions of Cl-, S-, Al- and alkali metal-containing compounds. Sodium and potassium chlorides can react with sulfur dioxide and aluminum silicate (the major component of the mineral and clay contamination) to form hydrogen chloride. Deposition of Cl-containing compounds and corrosion is particularly influenced by the aluminum silicate content and S/Cl ratio. If the S/Cl ratio is less than 2, there is a high risk of corrosion. When the S/Cl is higher than 4, the deposit does not cause corrosion. Recent studies show that the aluminum silicate/Cl ratio can dominate in its influence over the S/Cl ratio [31].

In order to prepare high-purity fuel, several methods have been suggested, such as washing the biomass with water [32].

Increasing grinding efficiency of lignocellulosic biomass

In recent years, significant progress has been achieved in increasing grinding efficiency of biomass [33], including the introduced mechanochemical approaches to the treatment of lignocelluloses of the plant raw materials [14, 34-38].

Preliminary treatment of the lignocelluloses materials of different morphology results in a decrease in their strength during grinding.

Destruction of lignocellulosic materials occurs mainly along the boundaries between different tissues, therefore, the particles that form consist primarily of cells of a single tissue. Further destruction proceeds to reduce the size of the particles. The density of cellulose (1.5 g/cm³) is 10-15% more than the density of lignocellulose. As we can see on fig. 1 the lignocellulose particles have the air bubbles in the structure in the place of cell core. When the particles are separated into fractions in cyclone equipment (fig. 2), the fractions show different values of the heat of combustion. Fractions of smaller particles rich in lignin demonstrate higher values of the heat of combustion (fig. 3). The content of lignin in the samples was between 6% and 12% .

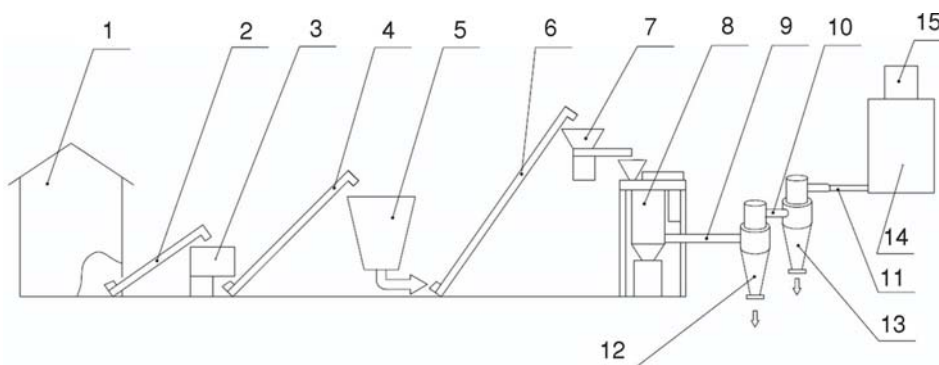


Figure 2. Technological line for the complex processing of lignocellulosic raw materials

1 – store of the raw materials, 2 – conveyer to transfer the raw materials into drier 3, 4 – conveyer to transfer the dried raw materials into preparation chamber 5 to pre-treat the raw materials before the mechanochemical operation, 6 – conveyer to transfer the pre-treated raw materials to feeder 7, 8 – mechanochemical mill, 9, 10, and 11 – pipes to feed the dispersed raw materials into separator 12, 13 – active filter; 14 – filter, 15 – fan

Cellulose and lignin show different intervals of thermal decomposition and different heat of combustion. Cellulose is oxidized at lower temperatures than lignin. Lignin is more stable to thermal destruction than polysaccharides and shows higher heat of combustion, which is related to its aromatic structure and an increased thermal stability due to the formation of new C-C bonds and condensed structures during pyrolysis. For this reason, the kinetic curves of oxidation of fractions differing in chemical composition show different behavior.

The best description of the curve for the sample containing 20% of lignin is achieved when 2 parallel reactions are considered described by the Avraami-Erofeev equations:

$$(E = 134 \pm 2 \text{ kJmol}^{-1}) f_1(\alpha) = (1 - \alpha)/[-\ln(1 - \alpha)]^{0.28} \text{ and}$$

$$(E = 155 \pm 4 \text{ kJmol}^{-1}) f_2(\alpha) = (1 - \alpha)/[-\ln(1 - \alpha)]^{1.22}.$$

The best description of the curve for the sample containing 70% of lignin is achieved when 2 parallel reactions are considered described by the equations: order equation ($E_1 = 132 \pm 12 \text{ kJmol}^{-1}$) $f_1(\alpha) = (1 - \alpha)^{1.3}$ and diffusion equation ($E_1 = 91 \pm 2 \text{ kJmol}^{-1}$) $f_2(\alpha) = 1/[(1 - \alpha)^{-0.33} - 1]$, fig. 4.

Direct experiments using a calorimetric bomb show that as the lignin content in the straw samples increases, the higher heating value of the material increases from 17.3 to 22.1 MJkg⁻¹.

It can be concluded that the experimental results and combustion models of biomass point to the necessity of decreasing the average particle size down to less than 100 μm . The powder fuel with higher lignin content has a high heating value and burns at a higher temperature. In order to increase the efficiency of grinding, the mechanochemical treatment should be carried out in optimized conditions in terms of duration and intensity.

Burning of the lignocellulosic powder fuel in cyclone and flare facilities

For burning particles smaller than 250 μm , cyclone set-ups have been suggested [39]. When such set-ups are designed, attention should be directed to the fact that biomass-derived fuel has to remain in the high-temperature zone for the time sufficient for the volatile components to evolve before the carbon residue starts reacting [40]. Laboratory tests show that burning under these conditions ensures low concentrations of carbon monoxide CO and carbon in the waste gas.

Research conducted in Russia on burning of coal powders is presented in the following publications [41-44]. Figure 5 shows a schematic of the test bed for studying combustion of powders [41].

Mechanical milling of coal was performed using a disintegrator-type free-impact mill or planetary roller mill. Burning of coal was studied in non-isothermal conditions of flaring.

The test bed is designed for the full technological cycle of burning of pulverized coal, gas fuel and has a prechamber of primary furnace, a shaft for coal re-burning, a system for gas cleaning and analytical equipment for analyzing the multicomponent gases and temperature measuring devices, which allow monitoring the process conditions in real time. The fuel is ignited during 1 min using a standard spark methane device or a plasma generator with a powder of up to 15 kW. The ignition device is then turned off such that burning of the fuel continues in an autothermal mode. Particles enter the flare in about 1 s after the milling operation has been completed.

Efficiency of lignocellulosic fuels in biotechnology industry

The efficiency of the proposed solution was calculated based on the energy parameters of the modern biotechnology processes of plant raw materials. An analysis was made for the technology of production of bioethanol from straw. A possibility was analyzed to cover the en-

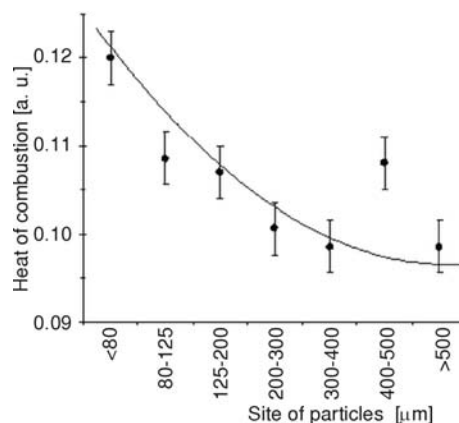


Figure 3. The dependence of combustion heat value (in arbitrary units) upon fuel particle size [24]

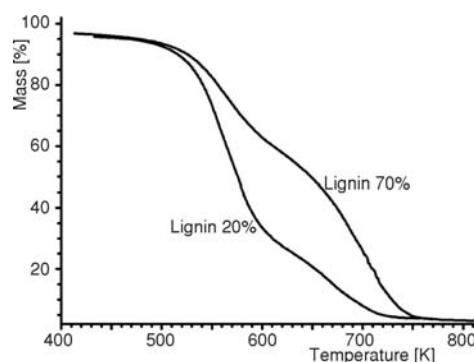


Figure 4. Thermogravimetry curves of 2 samples of straw (20% and 70% of lignin), particle size is smaller than 100 μm , heating rate 10 K/min (sample masses 5 mg, corundum sample holder, gas flow: oxygen 10 ml/min, argon 40 ml/min)

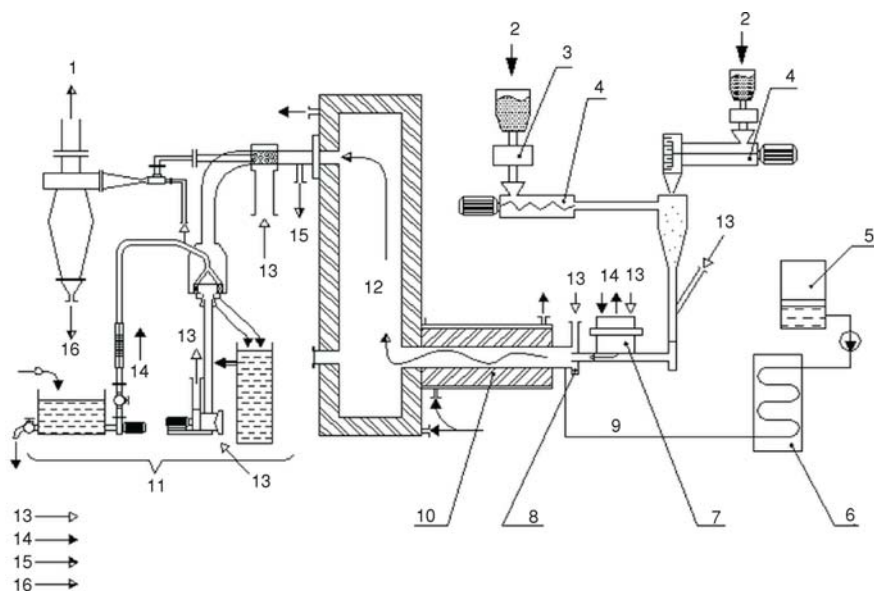


Figure 5. A schematic of the test bed for studying combustion of powder fuel

1 – waste gases, 2 – coal or biomass, 3 – feeder, 4 – mechanochemical mill, 5 – container, 6 – steam generator, 7 – plasma generator, 8 – cyclone-type burner, 9 – steam, 10 – primary furnace, 11 – fly-ash scrubber, 12 – boiler, 13 – air, 14 – water, 15 – gases, 16 – ash

ergy consumption by the technology used powder fuel obtained from the straw through grinding and concentration.

In the USA, the straw processing technologies are co-ordinated by National Renewable Energy Laboratory (NREL). The research results are available in the sources [45-47], from which the data given are taken.

The processing chain is a sequence of the following operations: «acceptance and storage of raw materials» → «pre-treatment of raw materials» → «preliminary hydrolysis» → «fermentation» → «distillation» → «storage».

Additional production lines are included: generation of electrical and thermal energy, growth of bacterial cultures, purification and preparation of water and production of the enzyme (cellulase) required at the fermentation stage. According to NREL, the contribution of the input energy to the cost of bioethanol is 18%. Therefore, it is important to include the generation of electrical and thermal energy based on utilization of waste materials into the main technological line.

Electrical and thermal energy are considered as by-products, which can be obtained by burning the solid residues of the distillation stage in a specially designed heat generator. The heat and electricity obtained cover the needs of the plant.

The production of steam consumes 49-50% of the energy released during burning of the lignin waste while 15.5% of this energy transforms into electricity. The elimination of the solution treatment stage from the technological line decreases the steam needs by 9.8-14.7%, which can increase the production of electric energy by 18.9-28.5%. This increase helps almost fully cover the electrical energy deficit for the process as a whole.

The data relevant to a biotechnological plant of NREL and necessary for the production efficiency calculations are:

- raw materials consumption – 675.3 thousand ton of straw per year,
- conversion of straw into ethanol – 292.6 liters of ethanol per 1 ton of straw,
- investment – 232.53 million USD,
- operational expenses – 1.522 USD/gallon of ethanol,
- productivity of the plant – 52.2 million gallon per year, and
- installed power of the equipment – 30470 kW.

The content of the carbohydrates in the straw used in the described technology is 40%, half of which will be separated to be used in biotechnology industry; therefore, the amount of the powder fuel available is approximately 540 thousand ton. An estimated calorific capacity of the powder fuel produced from plant raw materials is 7.0 MJ/kg.

The question of the complete electrical autonomy of the biotechnological plant should be discussed taking into account the presence of a power network in vicinity of the plant and the cost of electrical energy in this network. Other important factors are the cost of the electrical energy-generating equipment and a possibility of transferring the energy to external consumers.

The needs of the technology in steam are satisfied by its own capacities. In the NREL calculations, heating of the workplaces and welfare rooms is not taken into account as the plants are planned for regions with mild climate. Additional energy needs for heating of the workplaces and welfare rooms in Russia and Northern Europe can be also covered by the technology's capacities.

Conclusions

Destruction of plant tissues results in the formation of two types of the composite particles: one type primarily contains fragments of low-lignified cellulose cell walls while the other contains fragments of lignified cells. Particles differing in cellulose and lignin contents also differ in specific weight. Cyclone set-ups are capable of separating the powders produced by grinding of the lignocellulosic mass in a roller mill into fractions differing in density and enriched in either cellulose or lignin.

Mechanochemical treatment of lignocellulosic raw materials and separation of the ground product into cellulose-rich and lignin-rich fractions allows increasing the processing efficiency of plant materials by reclaiming the lignin waste and utilizing it as fuel in the same biotechnology industry.

The main developmental perspective of this work is the creation in the future of technology producing a lignified powder fuel for biotech companies. Energy consumption of modern biotechnology industries is such that the needs of an industry in steam and thermal energy can be satisfied by burning its own waste. Self-generation of electrical energy is possible; its efficiency depends on the costs of the energy-generating equipment and those associated with placing it in service and its maintenance.

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