Polymer and Biopolymer Analysis and Characterization

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POLYMER AND BIOPOLYMER ANALYSIS AND CHARACTERIZATION

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POLYMER AND BIOPOLYMER ANALYSIS AND CHARACTERIZATION

Preface

Chapter 1 - Particleboards are among the most popular materials used in interior and exterior applications in floors, walls, ceiling panels, cabinets and furniture. Particleboards combine low cost raw materials and simple processing. Decrease in raw materials and the need of conserving natural resources encourage the search for substitutes to replace natural wood in panel industry. Bio-based resources are renewable, widely distributed, available locally, recyclable, versatile, non-abrasive, biodegradable, compostable, and reactive. Rice husk is an abundantly available waste material in all rice producing countries. This residue has been used to produce a variety of added – value products, such as silica, biofuel and filler for composite materials. The present work is an attempt to report the state–of–the-art and future prospects of particleboards based on rice husk and also highlighting some results on the processing and characterization.

Chapter 2 - The past decades can be characterized by a rapid expansion in the use of polymers. Traditional materials, such as wood and metals, have been displaced in favor of lighter and stronger synthetic materials. The ability of melting and moulding polymers, combined with excellent physical properties, ensures that plastics will continue to grow in use and applications. Unfortunately, most polymers are not inherently stable to light or heat. During the service lifetime of a polymer, thermoxidative degradation occurs during polymerisation, storage, compounding, moulding of plastic articles and end use. Radiation, heat or shear lead to polymer chain breakage and the concomitant formation of free radicals, able to react with oxygen to form peroxy-radicals and these in turn scavenge a hydrogen atom from a second polymer chain [1]. Degradation may be associated to irreversible changes in the chemical structure of the polymer, which influences its physical and chemical properties, morphology, molecular weight, tensile strength, elongation at break and colour. The resistance of a polymer towards degradation depends on its chemical structure and the type and amount of any impurities. The generally established oxidation process is referred to as the Bolland-Gee mechanism.

Nowadays, polymer degradation can be interesting in order to decrease the amount of litter and to improve the environment protection, but degradation could result in a technological problem for the polymer processing and during the shelf life of the packaging, rather than an advantage. Oxidative degradation must then be avoided, and this can be achieved by using antioxidants.

Natural antioxidants show good performance in reducing the oxidation reactions in many common polymers. In particular tocopherols have shown their ability to protect polymers and to avoid the problems inherent to the use of synthetic antioxidants (i.e. migration of undesirable compounds to foodstuff causing contamination and loss of the material properties). In summary, the addition of natural antioxidants will give new possibilities to obtain more stable and sustainable materials with a broad range of applications.

Chapter 3 - The use of metallic powder as fillers is widely used due to intrinsic nature of the polymeric matrices. The addition of metallic powder allows an improvement on some electrical properties such as dissipation on static electricity. In this work we have prepared composites based on an ethylene vinyl-acetate matrix, characterized by high flexibility, in which we have incorporated metallic Zn powder. Composites with different compositions were subjected to a chemical treatment (simulating corrosion conditions) and a complete mechanical characterization of the composites was carried out to determine the influence of the corrosion conditions on final performance. We have investigated surface changes induced by the chemical treatment and its influence on adhesive properties. We have worked on the development and validation of a mechanical behaviour model based on dispersion of spherical particles (metallic filler) on a homogeneous matrix (polymer). As a general way, the addition of metallic Zn powder into an EVA matrix promotes a considerable decrease on mechanical properties regarding material cohesion (tensile strength and elongation at break) while stiffness increases. The chemical treatment simulating corrosion conditions does not promote significative changes on mechanical properties of composites but important changes in surface roughness, and consequently adhesive properties, can be observed.

Chapter 4 - The high consumption of plastics has generated a great amount of residues which is necessary to process. This fact has created the development of recovering processes that allow to separate and recycle the different plastics, despite this, it is usual to find the presence of some impurities in the material at the end of the recycling process. The presence of these impurities causes some changes on material behavior which requires a detailed study. The polymeric blends have been studied by several authors and frequently have culminated in theoretical models for predicting the behavior of the blend. Generally, these models have offered good results. The equivalent box model is one of the predictive models of mechanical properties most used. This model contains a parameter regarding the interfacial adhesion. Other concept that contributes some information about interaction between components of the blend is the solubility parameter which can also predict the solubility of the polymers that compose the mixture. The connection between both parameters allows to predict the behavior of the polymeric blend.

Chapter 5 - Adhesion interaction of the most widely distributed species of microscopic fungi: Aspergillus niger, Trichoderma viride, Penicillium funiculosum, Aspergillus terreus to surfaces of materials (polymers, metals) is studied. The force of adhesion interaction was measured by the method of centrifugal detachment. On the base of analysis of kinetic curves the macroscopic characteristics of adhesion micro-organism–metal surface were obtained. We show on the example of Aspergillus niger that stochastic nature of adhesion of microorganisms cells is caused by heterogeneity of support surface, heterogeneity of conidium sizes, and distribution of them by forces of adhesion obeys the Gauss law. We established that structure of cellular wall of microscopic fungi was changed in dependence on age, and change of force of adhesion interaction correlated with changes in cellular wall structure of microscopic fungi, and dominating role in strengthening of adhesion interaction was played by increase of albuminous components concentration in surface layer of cell.

Chapter 6 - The design of barbotage-rotation installation with different profiles of blades of the rotator has been created on the basis of modeling method. As a result of the complex of aerohydrodynamic research in the system "gas - liquid" carried out on the above unit certain recommendations on the improvement of the construction and operating conditions have been developed with a view to a rise in the effectiveness of dust removal.

Chapter 7 - Thermal degradation models are very useful in the prediction of the polymer response under certain temperature conditions. Traditional kinetic models are being questioned, since they show some drawbacks when applied to simple degradation processes. Considering the basic expression for polymer degradation processes, we have developed a new model and it has been validated by comparison with traditional kinetic methods. The proposed model is based on the consideration of polymer degradation processes as described by two consecutive reactions. The resolution of the proposed equations by this method is simple and gives a dependency of the conversion degree as a function of time and temperature which fits quite accurately a sigmoidal plot in a wide range of experimental data. A very good match between experimental results obtained by thermogravimetric analysis of poly(acrylonitrile-butadiene-styrene), ABS, and polycarbonate, PC, and data obtained by application of the proposed model was observed.

Chapter 8 - Investigations of water transport in polymer mixtures where as a consequence of partial components compatibility the influence of structural factors on mechanic, barrier and other exploitation properties is revealed especially notably take on special significance. In presented work we try to show how morphologic characteristics of polymer mixtures influence on water diffusion parameters and how the last ones correlate with exploitation characteristics of mixtures on the example of mixed compositions of: (a). nature polymer product of bacteria vital functions – poly(3-oxybutirate) [P3HB], and (b). synthetic polymers of various polarity (hydrophilicity).

Such investigations seem necessary for us for detailed description of ecologically compatible (bio)destructed systems functioning in water mediums.

Chapter 9 - There are no reliable methods of agreement between the chromatographic retention volumes of various substances and their physical–chemical properties at present time. That is why it is impossible to forecast these characteristics for unstudied substances. Well–known works in this field [1, 2] have either qualitative character or give the results suitable only for ranges of similar substances, predominantly for homologous series; the latter is caused by complicated character of the interactions taking place in system "stationary chromatographic phase – absorbed substance", which is impossible to describe adequately by an influence of one parameter.

Chapter 10 - Rigid polyurethane foams were prepared from a commercial system using clay as filler (0, 5, 10 and 20 wt.%). The effect of foaming process on the dispersion of clay in the polymeric matrix and their consequences on the physical properties of the final foams were investigated. The better dispersion of the clay was observed in the sample containing 5 wt% of clay by means of X-ray diffraction analysis and transmission electron microscopy (TEM). However, TEM photographs showed that in all the composites the clay layers were dispersed heterogeneously in the polymeric matrix. Regions with agglomerates, intercalation of the polymeric chain in the clay galleries and small amount of exfoliated clay sheets were observed. Also the analysis by TEM showed a slight alignment of silicate layers as a result of biaxial flow during the foaming process. The filled foams were characterized and the compressive properties were determined. The modulus and compressive strength showed a maximum for a clay content of 5 wt.%. However, an improvement in the thermal stability with the clay content was not observed.

Chapter 11 - With the use of ESR–spectroscopic investigations it has been studied the kinetics of bimolecular decay of methacrylic macroradicals in polymeric matrixes by networked and un–networked types. It was determined, that the macroradicals decay via the all range of temperatures (20 – 70 ^{0}C) is definitely ordered to the second order reaction kinetic equation. It were estimated the activate parameters of the process. With the use of Smolukhovsky's and Einstein's equations and also fractal dependence of conformation radius of the macroradical on its polymerization degree it was obtained the equation for the constant of bimolecular radicals decay in the diffusion regulations. This equation is in good agreement with the all set of experimental data and possesses by commonness permitting to use it for the analysis of bimolecular chain termination in different reactive zones via the polymerization processes ill the high conversions.

Chapter 12 - Mechanisms of interactions of nitrogen oxides with polymers of various classes are considered. Special attention is paid to analysis of mechanism of generation of stable nitrogen-containing radicals in reactions of nitrogen oxides with various functional groups of macromolecules. It is shown that in dependence on the nature of functional groups formation of stable radicals is conditioned by proceeding of both free-radical and ion-radical reactions. Application of reactions of nitrogen oxides for reception of spin-marked macromolecules and investigation of structure and molecular-dynamic properties of polymer materials are discussed.

Chapter 13 - Analysis of kinetic data is a necessary stage of investigation of processes mechanisms and predicting of practically important properties. Hard approach to such analysis is a traditional inverse problem of chemical kinetics solving with prescribed a priori system of kinetic equations. Alternative is so-called soft approach in which kinetic model is clearly not used. Both approaches have strong and weak sides. In this Chapter we compare these methods, consider methodological questions topical for analysis of kinetic data. Work is illustrated by two examples. In the first one we considered the technique of estimation of parameters of kinetic model. Hard method of Successive Bayesian Estimation (SBE) is used for reception of kinetic information from spectrum data in the case when spectra of "pure" components are unknown. It compared with various soft methods. In the second example the possibility of estimation of antioxidants activity by differential scanning calorimetry (DSC) is studied. Here we used the both hard approach with the help of non-linear regression analysis of data and soft approach based on the method of projection on latent structures in combination with novel method of simple interval calculation.

Chapter 14 - On the base of owned experimental and literature data we considered the free-radicals mechanisms of polysaccharides radiolysis products formation, i.e. compounds perspective for usage as additives to fodders for farming animals with the aim of increase of their sustenance and solution of utilization problem of polysaccharide-containing waste of agricultural production by radiation processing.

Chapter 15 - A quantitative generalization of the indices of swelling process of polymers, containing the non-hydrocarbon groups (polyurethanes, fluoropolymers) in organic solvents, as well as of the process of dissolution of poly(methyl methacrylate) was successfully realized by means of multiparametric equations, which take into account the solvents ability to specific and nonspecific solvation of polymers and solvents molar volumes.

Contents

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Chapter I

Particleboards Based on Rice Husk

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Abstract

Particleboards are among the most popular materials used in interior and exterior applications in floors, walls, ceiling panels, cabinets and furniture. Particleboards combine low cost raw materials and simple processing. Decrease in raw materials and the need of conserving natural resources encourage the search for substitutes to replace natural wood in panel industry. Bio-based resources are renewable, widely distributed, available locally, recyclable, versatile, non-abrasive, biodegradable, compostable, and reactive. Rice husk is an abundantly available waste material in all rice producing countries. This residue has been used to produce a variety of added – value products, such as silica, biofuel and filler for composite materials. The present work is an attempt to report the state–of–the-art and future prospects of particleboards based on rice husk and also highlighting some results on the processing and characterization.

1. Introduction

Agricultural by-products are emerging as new and inexpensive materials with commercial viability and environmental acceptability [1]. Among this kind of materials, lignocellulosic fibres are considered as attractive candidates to replace synthetic fibres in reinforced thermoplastics [2-7]. In this way, it is possible to obtain composite materials with properties similar to those of the already known synthetic fibre-reinforced plastics with the

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additional advantages of their low cost, low density, enhanced energy recovery, biodegradability and recyclability [1].

The manufacture of wood-based panels has caused an increasing interest in investigating ways and means of using trees more efficiently [8]. Wood composites can be made from wood waste or non-wood fibres. Agricultural residues are renewable resources that can be utilised as raw materials for particleboard manufacture. Research has been carried out on a wide variety of agricultural residues from many different regions of the world: wheat-cereal straws [9,10], shells and hulls [11-13], bagasse [14], and maize husk and cob [15].

One of the agricultural residues which can be potentially used as filler of polymeric materials is rice husk. Rice husk is a by-product of rice milling process, and a great resource as a raw biomass material for manufacturing added-value composite products. Presently, the world production of rice is approximately 500 million tons a year containing approximately 50–100 million tons of rice husk, 90% of which is generated in developing countries [16]. The main utilization of this source of biomass is in the production of ashes which are normally applied as additive for concrete [17-20]; as fuel [21,22]; to produce silica [23-26] and as reinforcing agent for thermoplastics [27-29]. Other and less explored route to revalorise this residue is as replacement of wood in particleboards [30-39].

The main objective of the present work is to report the state–of-the–art and future prospects of rice husk based particleboards.

2. Topography, Chemical Composition and Properties of Rice Husk

Generally, physical and mechanical properties of plant fibres depend on the cellulose content [39]. Indeed, the structure and properties of natural fibres depend on their source, age, etc. In the case of RH, the outer and the inner epidermis are different both, topographically and in cell composition [27].

SEM photographs of surface features and internal tissue of husk are provided in figure 1. The outer surface of lemma is highly rough and shows ridged structures (figure 1 b). The epidermal cells are arranged in linear ridges, which are punctuated with prominent conical protrusions. Figure 1c shows the inner epidermis of rice husk, which has a lamella structure. The morphology is different for the outer and inner surfaces of rice husk. Silica is mainly located in the tips of the protrusions and in low concentration, in other regions of the lemma. The outer epidermis is highly silificated with lignified fibres that provide strength, rigidity and stiffness to husk. Major components of rice husk are cellulose (25-35wt.%), hemicelluloses (18-21wt.%), lignin (26-31wt.%), waxes, amorphous silica (15-17wt%), absorbed moisture (8-11wt.%) and water soluble substances (2-5wt.%) [37,41]. The differences between the two surfaces of rice husk have to be considered together with other factors such as physical or chemical treatment, use of coupling agents and processing conditions when considering its use in composite materials based on rice husk.

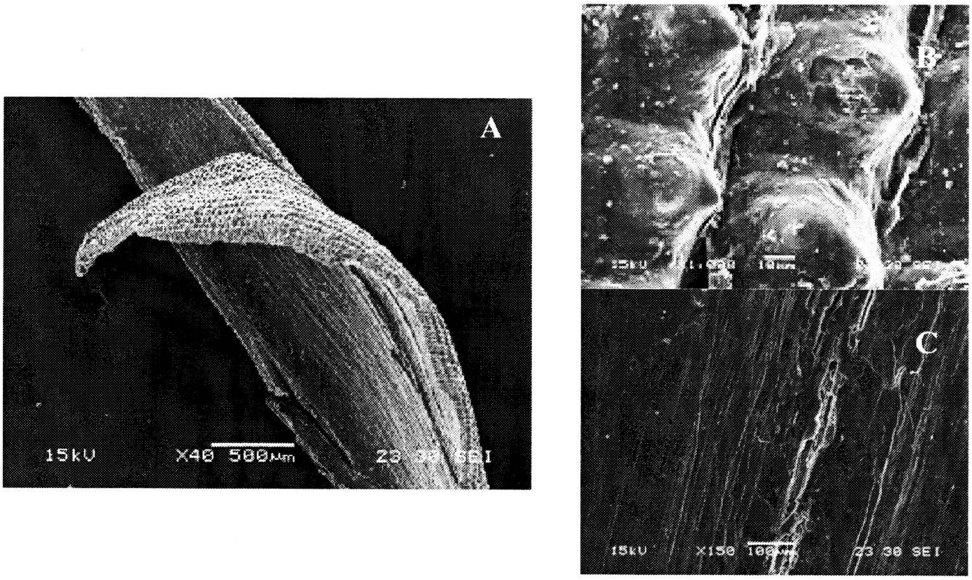

Figure 1. SEM photograph of rice husk (a), outer epidermis (b), and inner epidermis (c).

The presence of amorphous silica imparts effective resistance to water absorption process. The moisture content (on dry basis) of rice husk is in the range of 9-11 wt.% [37]. In a recent study, Abhay et al. [42] have analysed the liquid water absorption characteristics of paddy, brown rice and husk and have determined the diffusivity of liquid water transport at different soaking temperatures as well as their required activation energy. The diffusion coefficients for paddy, brown rice and husk at different temperatures followed an Arrhenius-type relation and the activation energy *(Ea)* was estimated using the E_a/R parameter of the Arrhenius equation. The activation energy of brown rice was approximately 1.2 times higher than the activation energy of paddy and 1.9 times that of the husk. This last difference can be attributed to the high silica content present in rice husk.

Despite the exhaustive studies carried out on various aspects of rice husk, limited information about physical and thermal properties is available. Mishra et al. [43] have reported the values of true density, thermal conductivity, contact angle and specific heat of ungrounded and grounded rice husk of Indian variety (Mashuri). The contact angle for ungrounded rice husk was higher than that of the grounded, may be due to the roughness of the surface which causes the inherent resistance to wet. The true density of ungrounded rice husk increases linearly with moisture content (from 1.021 to 1.054 g/cm^3 for moisture content from 11.4% to 21.3%).

It is important to remark that one of the main drawbacks in using rice husk as reinforcing agent of thermoplastics or in particle boards is the high silica content which decreases the interfacial adhesion between rice husk and the polymer matrix or the adhesive. To improve compatibility, silica can be removed or rice husk can be reacted with coupling agents, depending of the rice husk state (entire, fragmented or grounded).

3. Technical Applications of Rice Husk

Rice husk is a valuable natural resource not only as an excellent source of high quality silica, but also as a source of lignocellulosic material which can be potentially used to produce a range of valuable composite products. Technical applications of rice husk include: use in making cement and cementious materials [17-20], for fuel production [21,22], for producing silica [23-26], as reinforcing agent for thermoplastics [27-29], and for manufacturing particle boards [30-39]. Some of these applications are described below:

Component of Cementious Materials

Rodriguez de Sensale [17] reported the development of concretes with rice husk ashes (RHA). The addition of RHA provides a positive effect on the compressive strength of concretes at early ages, but in the long term, the behaviour of the concretes with RHA produced by controlled incineration was more significant. Rice husk is used to produce a light-weight concrete; as well as the pozzolanic effect which has a beneficial effect on durability, the fibres have the effect of improving the tensile strength of the material [18,19]. Ajiwe et al. [20] have developed economical cements from local and inexpensive raw material such as RHA. The major aspect of the project was to determine if the rice husk ash could be used for the substitution of silica in cement formulation so as to reduce its environmental hazard as a farm waste. The obtained cement showed comparable properties than those produced from clay and limestone. The production of cement from RH was relatively cheap and cost was comparable to that produced from the usual raw materials.

Fuel Production

Another increasing application of husk residues in rice producing countries, such as Brazil and Argentina, is as fuel in heat generation for drying operations, due to its high calorific power. The utilisation of this source of biomass through energy recovery would solve both a disposal problem and also generate useful energy. RH can be converted into liquid fuels by thermal conversion. The liquid fuel is a complicated organic compound that consists mainly of water, acids and heterocyclic substances. It can be used directly as a fuel oil for combustion in a boiler or a furnace without any upgrading. Alternatively, it can be refined for use as vehicle fuel [21]. In addition, pyrolysis of RH was studied under conventional conditions with the aim of determining the characteristics of the charcoal formed for its applicability as a solid fuel [22].

Silica Production

The major constituents of RH are lignocellulosic materials and amorphous silica (about 15-22 wt.%, depending on the rice type). Because the silicon atoms in the rice husk have been naturally and uniformly dispersed by molecular units, very fine particle size, with very high purity and surface area silica powder can be prepared under controlled conditions. Several reports can be obtained from literature concerning the production of silica from rice husk [23-26]. This process has the benefit not only of producing valuable silica powder, but also of reducing disposal and pollution problems. Approximately one-fifth of the ash is obtained on burning rice husk in air. The ash contains >90% silica by mass with minor amounts of metallic elements. Because the ash is obtained as a fine powder, it does not require further grinding, making it the most economical source of nanoscale silica [23]. Chandrasekhar et al. [24] have extensively analysed the research work on processing and characterization of rice husk ashes and reactive silica obtained as well as their applications. The silica produced by burning RH under controlled conditions is slightly coloured, amorphous and ultrafine in size and has high surface area and reactivity. White amorphous silica can be also produced by acid treatment followed by controlled burning. Carbon–free ash (white ash) was obtained from rice husk which opened a new perspective in its utilization concept, because high grade amorphous silica can be used for producing silicon and derivatives such as silicon tetrachloride employed in several kinds of industry applications [24-26].

Reinforcing Material

One of the most promising applications of rice husk is as filler for manufacturing lignocellulosic fiber–thermoplastic composites. The use of rice husk (entire or as flour) in making composite products, such as fiberboard and lignocellulosic fiber–thermoplastic composites is attracting much attention because of the potential for enormous gains in certain important properties of these products [27-29]. Intermolecular interactions between polymers are key factors in governing the strength and stability of composite interfaces. Cellulose fibres are very effective in forming hydrogen bonds with thermoplastic polymers, to produce composites with enhanced properties. However, in rice husk, cellulose is associated with hemicelluloses, lignin and silica [27], which reduces the bondability with polymers or adhesives. Thus, the attainment of good compatibility between rice husk and polymers or adhesives may be carried out by chemical or mechanical treatments.

B-A. Park et al. [27] have analysed in detail the effect of two different treatments on rice husk. Dry grinding produces rice husk fragments generated by mechanical force. These fragments retain the same surface characteristics than those of the entire husk. Differences between outer and inner surfaces are important factors for the performance of the composite products and must be considerate in order to use entire o fragmented rice husk.

The fractionation of lignocellulosics materials can be achieved also by steam explosion process (SEP) [27,37,44]. During SEP, rice husk is heated with saturated vapour in a pressure vessel during short time (56-180s) [27,37]. At the end of this period the release of pressure leads to an adiabatic expansion of the water present in the tissues [44]. Steam explosion is known to soften/melt lignin, in addition to removal of hemicellulose by hydrolysis.

Dry grinding produces segments of rice husks with predominating epidermal surfaces, meanwhile steam explosion separates rice husk into fibrous components [27]. From the practical point of view, exposed fibres would be the most suitable material for compounding with polymeric matrices while entire or fragmented husk are more suitable for particle boards or as reinforcing material in building materials [27].

Interfacial adhesion of cellulosic materials and adhesives or thermoplastics can be improved by treating this fibres with suitable chemicals. Many studies are reported in the literature concerning the chemical treatment of natural fibres, but little information is available for high-content silica lignocellulosic materials. Wheat straw is also an agroindustrial residue which is a potential alternative for some wood products. The presence of waxes and inorganic silica on the surface of straw reduces the bondability in the composite and they can be removed by bleaching. This method requires alkaline and oxidizing agents, such as NaOH and H_2O_2 at certain temperature [10]. Sodium hypochlorite in the bleach seems to remove more efficiently the wax layer and silica, producing a rougher surface than other chemicals. Hence, the enhancement in bondability may be obtained not only by exposing the hydroxyl groups but also by mechanical bonding between the lignocellulosic material and adhesives that penetrate easily inside the structure.

Another but less explored alternative is the use of coupling agents. The performance of composite products is determined by the structural, chemical, physical, and engineering properties of their individual components. Thus, the properties of composite products made from rice husks will be related to the characteristics of fragments and fibers derived from husks. For example, it has been observed that the use of a coupling agent improves the interfacial adhesion between rice husk flour (RHF) and thermoplastic matrix. Rice husks were subjected to dry-grinding and steam-explosion to reduce their sizes and subsequently, the surface of the particles was modified by using two different coupling agents, maleated polypropylene (MAPP) and γ-aminopropyltriethoxysilane to induce chemical reactions between the husk surface and the coupling agents [28]. It was observed that treated with MAPP, the O/C ratio of the husk surface decreased for both dry ground and steam-exploded husk. The treatment with silane resulted in an increase in the Si/O ratio for dry ground husk surface while this ratio decreased for steam-exploded husk particles. These results indicated that both coupling agents might be linked to the husk surface through chemical reactions. These results suggest that chemical characteristics of the surface of rice husk are crucial in developing the interfacial adhesion. Rice husk flour was also used as reinforcing agent of low and high - density polyethylene (LDPE and HDPE) using (MAPP) and maleated polyethylene (MAPE) as compatibilizing agents [28]. With the addition of the compatibilizing agent, the interfacial bonding between the filler and the matrix polymer was greatly improved, resulting in improved dimensional stabilities and water absorption behaviors.

Particleboard

Particleboard is defined as a panel product manufactured from lignocellulosic materials, primarily in the form of discrete particles, combined with a synthetic resin or other suitable binder and bonded together under heat and pressure. The primary difference between particleboard and other reconstituted wood products, such as waferboard, oriented strandboard, medium density fiberboard, and hardboard, is the material or particles used in its