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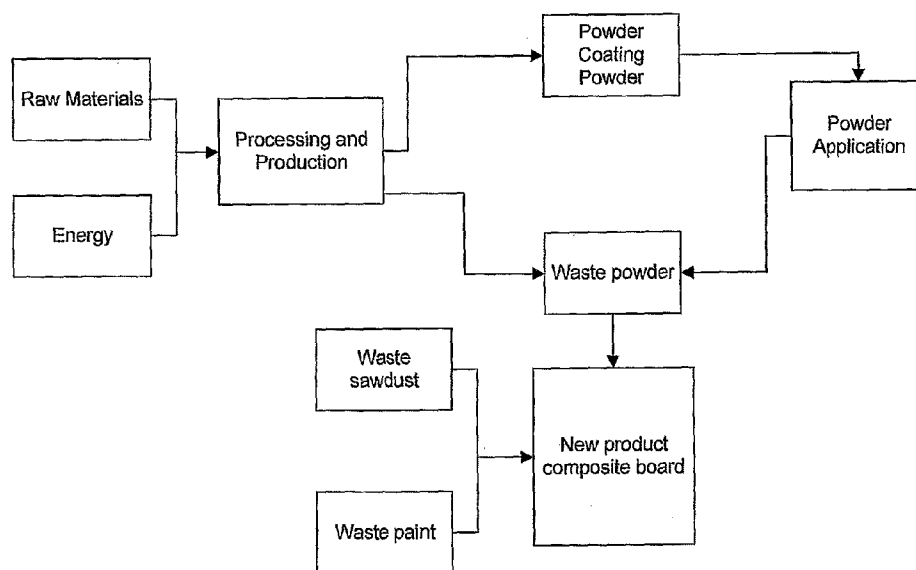
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(54) Title: COMPOSITE MATERIAL MANUFACTURED FROM A BINDER SYSTEM INCLUDING WASTE POWDER COATING POWDER



(57) Abstract: A process for manufacturing a water resistant board from wood fibre involves the use of a liquid binder system using waste powder coating powder (either a thermosetting resin such as polyester or epoxy or a thermoplastic powder) together with a water based acrylic paint. A typical composite board is made by mixing together from 15% to 30% of waste powder coating powder, typically a polyester thermosetting resin, with sawdust having a particle size typically below 3mm, there being from 40% to 75% of sawdust in the mix, and from 0.1% to 30% of a water based acrylic paint. All of the ingredients used to produce this board are typically waste materials, mixed in a low shear mixer, then moulded under heat and pressure, either continuously, or in individual moulds, to allow the thermoplastic and thermosetting resins to cure, and to encapsulate the sawdust particles to produce a waterproof composite board.

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COMPOSITE MATERIAL MANUFACTURED FROM A BINDER SYSTEM INCLUDING WASTE POWDER COATING POWDER

5 FIELD OF THE INVENTION

This invention relates to a product and/or method of manufacture. The invention is directed particularly, but not solely towards products made from waste materials.

BACKGROUND OF THE INVENTION

10 Waste material is a huge problem. For example worldwide over 1 million tonnes of powder coating powder (mainly thermosetting powders) is manufactured which may produce over 50 thousand tonnes of waste from the manufacturing process per annum. Even in New Zealand, approximately 150 tonnes of waste is produced from the manufacturing process. India produces over 650 tonnes and Australia more than 500 tonnes per year of waste
15 thermosetting powder material. The waste produced by the powder applicators worldwide could be as high as 250 thousand tonnes in addition to the waste produced from the manufacturing process. Because of the magnitude of the waste quantities involved no matter what one does, it is going to cost something. Even doing "nothing" means one has to allow for ongoing storage and handling.

20 Simply dumping the waste material also involves further handling costs and dumping charges. Additionally if the waste is toxic, extra costs will ensue. Thermosetting material has a chemical structure that it is not able to be easily economically reprocessed. Waste coatings such as paints can also be harmful to the environment and so makes disposal difficult for casual users and manufacturers.

25 Normally waste is produced in continuous quantities by large companies such as industrial concerns, where waste is either stored and/or dumped at the appropriate time. Today this cost is assumed to be a permanent overhead. Ultimately other ways are desired to reduce the production of waste, find a cost-effective way to dispose of it or the best solution would be to

reutilise it. Just the sheer size of waste produced also puts pressure on the landfills and their expected useful economic life. If the waste is toxic environmental concerns are another problem that must be faced. (For example, there is over 34 million litres of waste paint produced in USA per year and disposal of this is causing huge problems).

- 5 Powder coatings are popular because of their favourable environmental attributes and performance advantages. These powder coating powders can be either thermoplastic or thermosetting types. In powder coating, the finely ground particles containing resin, pigment and other additives are most likely electro statically charged and sprayed onto an earthed substrate and then heated so that the powder can melt, flow and cure. Typical particle sizes
10 are in the range 5 to 150 microns.

Powder coatings are available to the industrial finisher in two major types, thermoplastic plastic powders, and thermosetting plastics powders (often abbreviated to "thermosets").

Figure 1 is a schematic drawing of the differences between these two types of resins.

- Like all other industrial surface coatings, powder coatings are individually formulated to
15 meet the industrial user's very specific finishing needs. Thermoplastic powders consist of long molecules, each of which may have side chains or groups that are not attached to other molecules (i.e., are not crosslinked). Thus, they can be repeatedly melted and solidified by heating and cooling so that they can be reused. No chemical change generally takes place during forming. Usually, thermoplastic powders contain additives to enhance processing or
20 to provide necessary characteristics in the finished product (e.g., colour, conductivity, etc.). The service temperature range of thermoplastics is limited by their loss of physical strength and eventual melting at elevated temperatures. Thermoplastic powder coatings are based on thermoplastic resins of high molecular weight which help impart the physical properties. A thermoplastic powder coating melts and flows when heat is applied, but continues to have
25 the same chemical composition once it cools to a solid coating. These tough and resistant resins tend to be difficult, as well as expensive, to be ground into very fine particles necessary for the spray application and fusing of thin films. Typical examples of thermoplastic powder coatings are nylon, vinyl (PVC and fluoropolymers), and polyolefins (polyethylene and polyester). These powders are most suited for coating items requiring a
30 thicker film for extreme performance. They do not generally compete in the same markets as liquid paints.

Thermoset plastics, on the other hand, react during the curing process to form crosslinked structures that generally cannot be remelted and reprocessed. Thermoset plastic scrap must be either discarded or used as low-cost filler in other products. Thermoset plastic may be supplied in liquid form or as a partially polymerised solid. In their uncured condition, they can be formed to the finished product shape with or without pressure and polymerized by using chemicals or heat. Thermosetting powder coatings are based on lower molecular weight solid resins. Powders based on these systems can be ground to fine particle sizes with the average generally, in the range of 25-40 microns. When subjected to elevated temperatures, these coatings melt, flow and chemically crosslink within themselves or with other reactive components to form a higher molecular weight reaction product. The coating has a different chemical structure than the basic resin. Once cured, thermosetting powders are heat stable and, unlike thermoplastic powder coatings, will not revert back to the molten stage when reheated. Thermosetting powders are also tougher, have better adhesion to metal substrates, and are more resistant to solvents and chemicals than thermoplastic coatings. .Due to rheological characteristic of these resin systems, they can produce thin paint like surface coatings in the 1 to 3 mil (approximately 0.025 to 0.076mm) range with properties equivalent and sometimes superior to the coatings produced by the liquid compliance technologies. Examples of The types of resins used in thermosetting powders are- epoxy, polyester, acrylic, urethanes and silicone.

The future trends based on the chemistry of powder coatings are likely to be:

- Powders based solely on epoxy resin will continue to decrease.
- The share of powder based on polyester-epoxy will continue to increase, mainly for general industrial and indoor applications.
- Polyester and polyurethane-based powders will continue to grow due to demand for durable exterior coatings.

The use of TGIC-cured polyester will decline substantially due to the toxicity of TGIC. (Triglycidyl isocyanurate is a complex chemical used to crosslink, especially polyester powders)

- As a result the usage of alternative curing agents such as HAA will increase.

- Acrylic-based powders will be increasingly used for automotive applications.

Existing engineered panels or boards such as bison board (trade mark), particle board, oriented strand board (OSB) or MDF and various other composites have been developed utilising some waste material.

- 5 However existing engineered panel or board formulations may include toxic chemicals such as formaldehyde, which is used as an adhesive or glue. Formaldehyde is harmful to humans whereby it is known to cause various forms of building-type sickness in users and may be carcinogenic. Additionally, these engineered panels can be susceptible to water damage and do not possess adequate strength for some situations whereby they may fail in a sudden and
- 10 non-predictable manner.

DEFINITIONS

- Powder coating** - is a type of dry coating, which is applied as a free-flowing, dry powder. The main difference between a conventional liquid paint and a powder coating is that the
- 15 powder coating does not require a solvent to keep the plastics component and filler parts in a liquid suspension form. The coating is typically applied electrostatically and is then cured under heat to allow it to flow and form an impermeable film. The powder may be a thermoplastic or a thermoset polymer. Powder size can vary from 5 to 150 microns but is usually less than 50 microns. Average powder size is of a thermosetting plastics powder (the
- 20 majority of powders) is of the order of 30 to 40 microns.

- Curing** – when a thermoset powder is exposed to elevated temperature, it begins to melt, flows out, and then chemically reacts to form a higher molecular weight polymer in a network-like structure. This cure process, called crosslinking, requires a certain degree of
- 25 temperature for a certain length of time in order to reach full cure and establish the full film properties that the material was designed for. Normally the powders cure at 180°C in 8 minutes. The curing schedule could vary according to the manufacturer's specifications.

Primid - is a trade name for a HAA cross linker.

HAA - hydroxy alkyl amide.

30

TGIC - is an abbreviation of Triglycidyl isocyanurate;

MDF - medium density fibre boards;

MRGP - moisture resistant general-purpose boards;

HPPB - high performance particleboards

5 **Thermosetting powder coating resins include:**

Epoxy

Epoxy-Polyester Hybrid

Urethane Polyester

Polyester TGIC

10 Polyester HAA

Acrylic

Polyvinyl Chloride

Thermoplastic powder coating resins include:

15 Polyolefin

Nylon

Polyester

Polyvinylidene Fluoride

20 **PRIOR ART**

US Patent 3,671,615 by Price teaches "A method for making a composite board from scrap materials".

US Patent 5,215,625 by Burton discloses "A method for producing articles from waste fibre, waste/surplus paint, and waste/surplus ink".

25 US Patent 5,160,628 by Gerace et al discloses "A method of making filler from automotive paint sludge, filler, and sealant containing filler".

US Patent 7,022,756 by Singer discloses "A method of manufacturing composite board". This is concerned with the use of waste MDF boards that are chopped and milled into particles having a size between 20 mesh and 150 mesh to create waste flour, and then encapsulating that in a thermal plastic binder.

- 5 All documents or other material mentioned in this specification, including in particular any patents or patent applications cited in this specification are hereby incorporated by way of reference.

Documents identified herein have been located by hindsight searches (with knowledge of the invention) conducted by or on behalf of the applicant or located by patent examiners
10 conducting such searches with full knowledge of the invention set forth in the claims. The discussion of any document mentioned herein reflects what the author(s) of the document asserts, and the applicant reserves the right to challenge the accuracy and relevance of any of the documents mentioned.

It will also be understood that, although a number of documents may be mentioned; it is not
15 to be taken as an admission that any of these documents form part of the common general knowledge in the art, whether in New Zealand, or in any other country.

OBJECT

It is therefore one of the objects of the present invention to provide an improved product(s)
20 and/or method of manufacture which will obviate or minimise some if not all the aforementioned problems in a simple yet effective manner or which will at least provide the public with a useful choice.

STATEMENT OF INVENTION

25 In one aspect the invention provides a liquid or semi-liquid binder for use in the manufacture of a composite product containing a fibrous or particulate filler such as fibrous and particulate virgin or waste materials, such as sawdust, fly ash, plastic chips and shreds, plant and animal fibres and hair; wherein the liquid binder contains a proportion of a plastics powder coating powder comprising either a cross linkable thermosetting plastics powder or a

thermoplastic plastics powder or a mixture of such powders, which may or may not be suspended in a liquid carrier.

Preferably the liquid carrier is waste paint.

5 The binder can be manufactured prior to adding the filler, or can be made in situ by mixing the liquid paint with the sawdust and then later adding the powder, or by mixing all three ingredients at the same time, or in some other sequence. In some situations the binder may be mixed from the waste paint and waste powder and sold or delivered to a manufacture of composite materials.

10 In another aspect the invention provides a composite material containing from 10% to 80% by weight of a fibrous or particulate filler and a liquid binder, wherein the filler is chosen from the group comprising fibrous and particulate waste materials, sawdust, plastic chips and shreds, plant and animal fibres, waste or virgin materials: wool, plastics, rubber, fly ash, glass, sand, polyethylene, polyester fibre, and wood.

15 In another aspect the invention provides a composite material comprising a plurality of particulate or fibrous materials encapsulated within a plastics matrix wherein the plastic matrix consists predominately of a thermosetting resin but includes a percentage of thermoplastic resin.

Preferably the plastics matrix comprises from 10% to 60% by weight of the composite material.

20 In another aspect the invention provides a method of manufacturing a composite product from the following ingredients: a fibrous or particulate filler and a liquid binder which may or may not contain a proportion of a plastics powder coating powder comprising either a cross linkable thermosetting plastics powder or a thermoplastic plastics powder or a mixture of such powders, which may or may not be suspended in a liquid carrier wherein the method
25 includes the following steps of:

- (a) Mixing the ingredients at a low shear to form a mouldable mixture
- (b) moulding and curing the mixture to form a solid matrix.

Preferably the amount of thermosetting plastics powder or thermoplastics powder (or combination of the two) is from 10%-60% by weight based on the total weight of the resultant composite material, the liquid carrier is from 0.1% - 50% by weight based on the total weight of the composite material and the filler is 10% to 80% by weight based on the
5 total weight of the composite material.

Preferably one or more of the individual ingredients is recycled or waste material.

Preferably the liquid carrier comprises waste paint.

In yet another aspect the invention provides a composite material made from the application of heat and pressure to a mixture containing: (a) a filler chosen from the group comprising:
10 fibrous and particulate waste materials, sawdust, plastic chips and shreds, plant and animal fibres, waste or virgin materials: wool, plastics, rubber, fly ash, glass, sand, polyethylene, polyester fibre, and wood; (b) plastics powder coating powder comprising either a cross linkable thermosetting plastics powder or a thermoplastic plastics powder or a mixture of such powders, suspended in a liquid carrier; and (c) wherein the liquid carrier is chosen from
15 the group comprising: liquid paints, water based acrylic paints, solvent based acrylic paints, solvent based resin paints, waste paints, and water based surfactant mixtures.

The inventive step is the use of a waste coating powder (either a thermosetting resin such as polyester or epoxy or a thermoplastic powder) together with water based acrylic paint to provide a novel binding system for the manufacturing of a water resistant composite material
20 containing wood fibre or other particulate fillers.

A typical composite board is made by mixing together from 15% to 30% of waste coating powder, typically a polyester, epoxy, polyester-epoxy, polyurethane or acrylic thermosetting resin, with from 0.1% to 30% of a water based acrylic paint and this may be mixed to form a liquid binder or be mixed in separately with sawdust having a particle size typically below
25 3mm, there being from 40% to 75% of sawdust in the mix. All of the ingredients used to produce this board are typically waste materials, mixed in a low sheer mixer, then moulded under heat and pressure, either continuously, or in individual moulds, to allow the thermoplastic and thermosetting resins to cure, and to encapsulate the sawdust particles to produce a waterproof composite board.

In another aspect the invention may broadly be said to consist in a composite product(s) including the following ingredients, of a proportion of a substantially cross linkable thermosetting plastics material or a thermoplastics material and a proportion of a binder system which may include a diluent and additives mixed or combined such that the product
5 is formed.

Preferably the product is a liquid binder.

Preferably the product includes a bulk filler material which is capable of forming a matrix for the thermosetting plastic, thermoplastics or binder system materials to bind to and provide strength and flexibility to the matrix.

10 Preferably the bulk filler material is capable of binding to the thermosetting plastics, thermoplastics or binder system materials to form the matrix with the desired properties.

Preferably the cross linkable material is a thermosetting plastic material, thermoplastics or binder system in a solid or liquid form.

15 Preferably the bulk filler material is in a powder, flake, chip, fibre, shredded, comminuted or strand form.

Preferably the thermosetting plastic, thermoplastics or binder system is a waste material.

Preferably the bulk filler product is a waste cellulosic material.

Preferably the product includes a mixing agent wherein the mixing agent is water/solvent.

20 Preferably the product includes a wetting agent/dispersant sufficient to ensure homogenous mixing of all the ingredients.

Preferably the product includes an adhesive-like binder system to bind the material together.

Preferably the wetting agent/dispersant can include a binder system.

Preferably the binder system includes a hardener to form a cross-linked matrix which can be in a substantially liquid or solid form.

Preferably the bulk filler can be selected, for example but not exclusive and is selected from any one of waste or virgin materials, such as wool, plastics, rubber, fly ash, glass, sand, polyethylene, polyester fibre and wood.

Preferably the wetting agent can be a surfactant.

- 5 Preferably the product comprises about 10-60%, and more preferably about 30% thermosetting plastic or thermoplastics material, about 40-75% bulk filler product and about 20% of a binder system, for example paint.

Preferably the thermosetting plastics or thermoplastics powder can be in the range of 15-30% by weight.

- 10 Preferably the bulk filler product can be in the range of 50% by weight.

Preferably the binder system can be paint in a liquid form.

Alternatively the binder system can be paint in a solid form.

- 15 In a further aspect the invention may broadly be said to consist in product including the following ingredients, of a proportion of a material including a cross linkable thermosetting plastic or a thermoplastics non liquid material, a liquid binder system which may include a diluent and additives, and a proportion of bulk filler material mixed together or combined such that a product is formed.

- 20 In another aspect the invention may broadly be said to consist in a method of producing a product, the product including the following ingredients of a cross linkable thermosetting plastic or a thermoplastics material, bulk filler material and a binder system, wherein the method includes the following steps of:

Step 1: Mixing the ingredients (which may be mixed in a ribbon mixer) to form a mixture. (Some drying or hydration may be required at this stage)

Step 2: Placing the mixture in a mould(s).

- 25 Step 3: Pressing the mixture within the mould(s) to form the size, thickness and shape desired.

Step 4: Placing the mould(s) in a vessel for a set pressure, set temperature and a set time.

Step 5: Removing the mould(s) from the vessel.

Or alternatively

5 Step 1: Mixing at low shear the ingredients which may be mixed in a ribbon mixer to form a mixture (some drying or hydration may be required).

Step 2: Place the mixture in a continuous line/vessel where pressing into the desired size, shape and/or thickness and the line speed, pressure and temperature are predetermined.

Step 3: Remove end product from the vessel

10 The embodiments of the invention will now be described by way of example only, with reference to the drawings and the detailed description.

DRAWINGS AND THE DETAILED DESCRIPTION

15 Figure 1 illustrates the structural difference between (a) thermoplastic resins and (b) thermosetting resins used for powder coating.

Figure 2 an overview of the process for using waste powder coating powder (WPCP) to manufacture composite boards.

Figure 3 is a schematic of the process steps used to produce composite boards from waste powder coating powder (WPCP).

20 Figure 4 shows the chemical formula of TGIC cured powder with hydroxyl group.

Figure 5 shows the chemical formula of HAA cured powder with no hydroxyl groups.

Figure 6 shows the effective varying density against thickness swell of the resulting composite board.

- Figure 7 shows the effect of varying density against modulus of rupture for the resulting composite board.
- Figure 8 shows the effect of mixing method on the modulus of rupture of the resulting composite board.
- 5 Figure 9 is a photomicrograph of the composite board structure from fracture depth analysis, showing that the resin encapsulates the individual wood elements in a continuous matrix.
- Figure 10 is a photomicrograph showing a 3D picture of the fractured surface of the composite board.
- 10 Figure 11 shows two scanning electron micrographs of the fracture surfaces resulting from (a) internal bond strength test; and (b) a three point bend test.

DETAILED DESCRIPTION

The product of the invention can be formed into any desired product, e.g. panels, boards, battens, blocks, architectural mouldings and the like. As the process involves a liquid slurry or mouldable paste like stage, the products can be pressed into suitable moulds, extruded, or moulded and cured in a continuous or step-by-step fashion. Other non limiting examples include various building boards or sheets, pin boards, partitions, flooring, laminates, pallets, flower pots, door handles, dunnage, furniture, veneer boards, sign boards, acoustic insulation, thermal insulation, ceiling tiles, architraves or soffits .

The product includes the following components or ingredients, including a thermosetting plastic or thermoplastics material and/or binder system. The cross linkable material or thermoplastics material can be in or be formed as a non liquid form. In most cases it is preferable that the thermosetting plastic or thermoplastic material is a powder or mixture of powders. Typically it will be a waste powder from the manufacture or use of a powder coating process, although non-waste powder coating material could be used.

The binder system can be in a liquid form or be formed into a liquid form. This product combination of thermosetting plastic or thermoplastic material or a mixture of these powders

and the binder system, for example paint can be formed into a new binder product. This new binder product can be combined with any type of bulk filler to form another product. The cross linkable material can be for example a thermosetting plastic powder typically a polyester powder or a proprietary mix of thermosetting powders.

- 5 Alternatively to the cross linkable material there is thermoplastics material which can be in the form of a powder and the bulking filler material can be an organic or inorganic product. For example the organic product can be a wood product.

The thermosetting plastic or thermoplastics material can include various chemistries and particle sizes from any manufacturing plant, whereby the particle size can be, for example
10 35um (microns) on average or 'fines' which can be less than approximately 5um average in size.

The bulk filler material can include any particle size, which can then be reworked to suit if necessary. Some examples for bulk filler material sources are the following wood and wood composite waste by-products sanding dust, fibres, fluff and chips. It is also possible to use
15 waste material in the form of chips, fibres or 'fluff' from MDF boards which may contain urea-formaldehyde as this process can be used to encapsulate the formaldehyde within the resulting matrix and thus minimise the release of the formaldehyde. It is preferable that the filler is a hydrophilic material such as natural fibres, paper, wood fibres, sawdust, and wood chips.

20 The waste powder coating powder is hydrophobic or hygroscopic but when combined with a water based paint it becomes intimately bound with the hydrophilic fibres of the filler, and when the mixture is heated the waste powder melts and binds the fibres together into a sold matrix. Preferably the powder contains or consists predominately of a thermosetting plastic resin (examples include polyester and epoxy resins and blends thereof).

25 Functionally the cross linkable thermosetting plastic material sets permanently when heated and acts as the primary 'adhesive' to 'glue' the bulk filler products together to ensure the physical properties of the resulting product are maximised. The thermosetting plastic powder takes part in the cross-linking reaction to bind the matrix together and also acts as an adhesive to bind the matrix together, adds water-resistant qualities, is thermosetting and so
30 melts with heat and cures at approximately 180°celsius and so, for example can be processed

in a normal MDF manufacturing method to produce a MDF-type board. Once cured the powder is not able to be re-melted, so if subjected to heat will continue to hold the matrix together. Preferably the cross linkable material is stable when exposed to heat after the matrix is formed and must be compatible with the other ingredients or additives in the product formula to form the matrix with the desired properties.

Cellulosic material or wood products are one example of functioning to provide a matrix or bulk for the thermosetting plastic or thermoplastic powder and any other additive(s) to bind to. Additionally the wood products are used to provide strength and bulk to the matrix and also provide some flexibility. Minimally the cellulosic product must be compatible with and be able to bind to the thermosetting plastic or thermoplastic material and any other additive to form the matrix with the desired properties.

The product can optionally include a wetting agent/dispersant or mixing facilitator which can assist in mixing the ingredients together to form the matrix, thereby ensuring a homogenous mixing of all the ingredients. The wetting agent can also act as a dispersant for the thermosetting plastic or thermoplastic material. For example, the wetting agent can merely include water (and/or a solvent or liquid or solid) or more generally the wetting agent can be a binder system with or without a hardener but having some form of curing mechanism. The binder system, with a curing mechanism, can form a matrix with the bulk filler to give the desired properties. The binder system can include any suspension of a solid or solids in liquid which cures or cross links by evaporation and/or oxidation and may contain additives or pigments. The binder system may be water or solvent based or any combination thereof. We have found that waste water based acrylic paint is able to be handled with minimal health, safety and environmental concerns and is readily available.

Depending on what physical features are required many different formulations for the product are possible:

Example 1:

Thermosetting plastic or thermoplastics material 30% by weight,

Bulk filler product 50% by weight, and

Liquid carrier, e.g., liquid paint 20% by weight.

However when the paint or liquid carrier is not used, the following formulation for the product can be made up of:

Example 2:

Thermosetting plastic or thermoplastics material 30% by weight and,

5 Bulk filler product 70% by weight.

In general, each of the ingredients as indicated in the above examples has a range of percentage values by weight whereby their inherent properties are best utilised. The ranges are approximate and refer to desired properties and are not limiting.

10 In summary, each of the ingredients has the following ranges of best use for optimal properties though other percentages outside these ranges will still work:

1. Thermosetting plastic or thermoplastics material 15 to 30%;
2. Bulk filler product 40 to 75%;
3. Binder system, e.g., liquid paint 10 to 30%.

15 A minimum of approximately 15% of thermosetting plastic or thermoplastics material is required so that the cross-linking adhesive/binding and water resistant properties are obtained. Below 15% the product can become brittle and is less water resistant and loses its mechanical properties. Beyond the maximum of approximately 30% of the thermosetting plastic or thermoplastics material, the product can lose some of its elasticity and flexibility properties. This is due to the excess thermosetting plastic or thermoplastics material
20 particles adhering to each other in greater numbers rather than to the bulk filler products to cause a loss in mechanical properties.

The bulk filler product for example can be a cellulosic material such as a wood product or a packaging material that is treated or untreated (as the wood product is encapsulated by the paint and powder mixture it is possible to dispose of toxic treated sawdust containing copper,
25 chrome arsenic (the CCA treatment) . It can come from a variety of trees. It can also be sourced from virgin wood or from composite wood-based products such as MDF or particle board. It can also comprise a variety of particle sizes and shapes, depending on what type of

end product is required. Advantageously the cellulosic material can be sourced from waste products, whereby it has been found that treated waste wood product has been very difficult to recycle and therefore happens to be in plentiful supply. Untreated waste wood can also be used. However any source of wood can be used that is compatible with the chemistry of any
5 of the other ingredients.

If the bulk filler product content is below 40% the mechanical properties of the product are reduced somewhat whereby the product becomes brittle. If bulk filler content is greater than 75% there can be insufficient binder system containing the thermosetting plastic or thermoplastic material and liquid carrier, e.g., paint to act as an adhesive to hold the matrix
10 together, whereby the product can lose some of its desirable mechanical properties to become brittle and crumbly.

The liquid carrier in the binder system for example paint improves the cross-linking adhesive and water resistant properties of the end product, for example a flat board. It also acts as a wetting agent and dispersant to ensure all of the ingredients are thoroughly mixed to obtain a
15 better performance of the product. If the liquid carrier in the binder system, for example the paint by percentage weight is over 30% this tends to cause problems during the manufacturing process when heat pressing, with any water or solvent egress causing stress cracking in the product. Many variations, additives and sources of material ingredients are possible. It has been found that waste thermosetting plastic powder and waste cellulosic
20 material and waste liquid paint are ideally suited for manufacture into, for example an engineered product such as a particle board or MDF-type product.

Thermosetting plastic powder can easily be sourced new or from waste which cannot be economically reworked through standard manufacturing processes and or cannot be sold as a readily usable end product. The thermosetting plastic powder or powder coating material
25 can be found via any coating manufacturer and from powder applicators whereby waste is generated by overspray which does not adhere to the article being coated. There can also be waste powder generated from filters in filter systems. Thermoplastic plastic powder products can also be used in place of or in combination with thermosetting powders to assist in allowing the processing into various different articles. Other examples of thermosetting
30 powder types can be polyester, epoxy, polyurethane and acrylic which have good binding to

wood products, adds water resistance properties, is a thermosetting material and have good wetting properties to aid in uniform matrix formation.

Wood products can be swapped for or combined with other additives or alternatives such as wool fibres, shredded rubber, shredded plastics (e.g. polyethylene), glass, sand, fly ash, and
5 fabric such as polyester. These can be sourced from waste products.

A wide variety of hard or soft composite materials can be made using this process, by varying the percentages and types of ingredients. We prefer to use a liquid carrier (preferably a waste paint) but there may be some composite materials that could be made without the liquid carrier. Our tests show that such "dry mix" composites are inferior to the "wet mix"
10 composites. We have included an example of a "dry mix" composite. However, the liquid carrier aids in the wetting and disbursing of the waste thermosetting or thermoplastic material and bulk filler products and produces a far superior product. The mixing process is an important processing step whereby all ingredients should be thoroughly mixed to obtain maximum mechanical properties. The liquid carrier of the binder system, for example paint
15 also provides additional cross-linking, adhesive and water resistant properties to the thermoplastic plastic or thermosetting material.

Alternatively a solid binder system can be used instead of a liquid binder system, whereby during the manufacturing process extra steps could be included to alter the solid to form a liquid. Alternatively surfactants can be used in place of or in addition to the paint used as the
20 liquid carrier in the binder system. A surfactant provides wetting and dispersion properties so that the thermosetting or thermoplastic material can mix thoroughly to attain maximum cross-linking, adhesive and water resistant properties.

In another less preferred variation just a mixture of the bulk filler product and binder system, for example paint can produce another form of end product. Also included are further
25 examples (labelled 'Example 3', 'Example 4' and 'Example 5') of processes or methods of manufacturing the product:

Example 3:

A processing method and apparatus used to make a board:

Step 1: Cross linkable material (for example thermosetting powder) and binder system (for example liquid paint) are mixed with the bulk filler (for example wood products).

5 Step 2: The resulting mixture which may be mixed in a low shear mixer such as a ribbon mixer to form a mixture (some drying or hydration may be required) which can then be placed in a mould.

Step 3 The mixture in the mould can then be pressed into any shape desired, such as a flat sheet. This can be carried out in a cold press at a set pressure for a set time.

10 Step 4 The resulting moulded board is removed from the cold press and placed in a hot press at a set pressure and temperature for a set period of time.

NOTE – Step 3 may be eliminated and just the hot press used with varying pressures and/or temperatures. This is to reduce the entrapped air and allow vapours to escape to reduce the likelihood of stress cracking when the pressure is released. Also a multi opening daylight press could be used with step 3 included or eliminated.

15 For thicker boards, more of the mixture can be added after the first cold press whereby the cold press is pressed again. Also at this stage a veneer of wood paper or cardboard can also be added. In other variations of this method the hot press can be used simply in place of the cold press step.

Example 4:

20 The following is another example of a processing method and apparatus used to make a moulded product:

Step 1: Thermosetting powder and liquid paint is mixed with bulk filler products, which may be mixed in a ribbon mixer to form a mixture (some drying or hydration may be required).

25 Step 2 The resulting mixture is then placed in a mould of any desired shape. The mould is heated in an oven at a set temperature for a set time.

Step 3: The mould is allowed to cool, with the shape being removed from the mould.

Example 5:

A processing method and apparatus used to make a board:

Step 1: Mixing the ingredients which may be mixed in a ribbon mixer to form a mixture (some drying or hydration may be required).

5 Step 2: Place the mixture in a continuous line/vessel where pressing into the desired size, shape and/or thickness and the line speed, pressure and temperature are predetermined.

Step 3: Remove end product from the vessel

Experimental Studies:

10 For our study we used sawdust of Radiata Pine as the reinforcing matrix. Wood particles used were generally from treated wood but for some untreated wood was used. This was carried out to see if the treatment had any effect on the binding of the composite. The preservative treated saw dust was collected from a local saw mill which primarily cut lumber treated to the H3 and H4 standard grade with copper chromium arsenate (CCA).

15 The particle size of the sawdust used for most of this study was less than 3 mm in which about 50% was in the range of 1 to 2 mm. This is for either the untreated or the treated sawdust. For the study the wood particles were air dried to maintain moisture content of about 4 to 6% of the oven dry weight. Only in one study there were wood shavings used that had a much bigger particle size. This was specially to see if the particle size had any effect
20 on the mixing and binding action.

Water based acrylic paints have been used commercially for a number of years worldwide, and their superior performance for long term decoration and protection is well proven. It is a fast-drying paint containing pigment suspended in an acrylic polymer emulsion. This polymer emulsion is made of tiny spheres of the acrylic polymers held in suspension in a
25 medium of water. Acrylic paints become water-resistant when dry but do not become hard and rigid. The acrylic chemistry is very similar to that used in acrylic plastic sheet and moulding powder.

Acrylic resins for decorative paints are chemically designed to maximise resistance to weathering without being too hard or rigid for use as paints. An acrylic emulsion is a two-phase system in which acrylic polymer droplets are dispersed in an external water phase. Acrylic polymers, used as coating binders, are comprised chiefly of esters of acrylic and methacrylic acid that are polymerized by addition polymerization, usually using a free radical mechanism. By altering the ratios of the monomers, a chemist can change a polymer's balance of properties. Pure acrylic latexes (Jewell) are based on methacrylate ester monomers, once again copolymerized with esters of acrylic acid. Methyl methacrylate-2-ethylhexyl acrylate copolymer and Methyl methacrylate-butyl acrylate copolymer are two such examples.

The good weathering resistance of acrylic polymers is primarily due to their resistance to hydrolysis and their lack of absorption of ultraviolet (UV) light. This high-energy portion of the light spectrum is mainly responsible for degradation.

The acrylic emulsion contains tiny spheres of polymers held in suspension in water. When a thin film is applied to the surface, the water evaporates and the spheres are forced together by capillary pressure. After the water and coalescent have evaporated from the film the surface softens, the spheres fuse together to form a continuous film of polymer, which is then insoluble in water. The presence of coalescents helps to keep the surface of the spheres soft which in turn help to form a smooth film. Acrylic paints, to some degree, allow excessive moisture to escape in the form of water vapour. If the substrate is excessively wet then it can cause paint film failures due to poor adhesion. Further the evaporation of moisture will exert pressure on the film which may lead to blistering of the film.

When people buy paint for use there is always some quantity which is excess and it eventually ends up as waste which is difficult to dispose of safely. An EPA study Editorial May 2007 coatings world, p 6, shows that millions of gallons (65-69 million gallons in US alone) are available for safe disposal, reuse, or recycling. Some companies over the world (e.g. Dulux in Australia and Resene in New Zealand) have started to have the recycle scheme up and running.

Paint used for this project was Dulux Weathershield X 10 grade which was exterior use semi gloss acrylic. The colour used was Vivid white 529-04912 and was from 10 litre plastic

pails. The paint has an approximate weight per litre (WPL) of 1.289 kg and %solids by weight of approximately 53.42 (so water is 46.5%) with a % solid by volume being 40.0.

Approximate Paint composition is detailed in table 1

Ingredient	Percentage
Water	9
Propylene glycol	4
Dispersant	1
Antifoam	0.5
Surfactant 1	0.2
Ammonia	0.1
Titanium Dioxide	18
Calcium Carbonate	8
Zinc Oxide	0.2
In-can and dry film preservatives	0.5
100% acrylic latex	50
surfactant 2	0.1
coalescing agent	2
thickener 1	0.1
thickener 2	1
thickener 3	0.4
Pigments are added to make a mill-base under high shear in a high speed disperser.	

5 Composite Developments:

Composite particle boards were made with saw dust, paint, and waste powder coating powder. The powder and paint is the binder system and the saw dust is the reinforcing fibres.

Wood is known to be hygroscopic. When the saw dust is dried to a low moisture content it is more vulnerable to absorb moisture when placed in a moist condition. The powder coating powder is known to be hygroscopic as it absorbs small amounts of moisture on storage. The paint which is used in the composite manufacture is water based having about 50% water.

5 When all these three components are mixed together the paint is absorbed by both the saw dust as well as the powder coating powder. This in-turn helps link the powder and paint to the saw dust. In the designed composite manufacture there is heat and pressure that is simultaneously applied. During this process it is expected that the saw dust gets coated and then impregnated with the powder and paint.

10 The manufacturing process used for the particleboards is outlined below in a schematic diagram shown in Figure 3.

The approximate densities of the three raw materials are as follows:

- Density of sawdust = 150 kg/m^3
- Density of powder = 1310 kg/m^3
- 15 • Density of paint = 620 kg/m^3

Mechanical mixing or blending of raw materials:

The way in which the powder/paint binder system is added to the cellulosic particles is of great importance as the board properties heavily rely on the adhesion between the cellulosic fibres and the powder/paint binder system. A homogeneous mix will ensure that all the raw materials are uniformly dispersed. This is desirable because the dispersion of the powder/paint binder system through the cellulosic particles is important to bring about good adhesion. Mixing has to blend the materials with the particle size of the cellulosic particles not be affected. If the mixing action is harsh that is if there is high sheer then there will be good dispersion but the particle size of the cellulosic filler (sawdust) is bound to reduce. This was the main concern in deciding the correct equipment for mixing. Hand mixing was tried in the initial laboratory trials as it could be controlled but this is not a process that can be used commercially. The mixaco mixer that was used was found to reduce the particle size due to a higher sheer rate. The melt mixing in an extruder was found to somewhat bring about partial precure that affected the final curing step. The ribbon blender was seen to be

20

25

most suited for the mixing process as these results were seen to be the best. The quantities that can be mixed at one time to can match with commercial production targets.

Ribbon Blender Mixing:

Ribbon blenders are ideal for uniform batch mixing where time is not a constraint but having the advantage of lower investment with a larger batch size. This was specially acquired and developed to be used for mixing about 50kg's of the raw materials at one time. The order in which the raw materials were introduced in the blender was the saw dust first and over that the paint. This was allowed to mix for approximately 10minutes and then the powder was poured while the mixing was uninterrupted. Further mixing was carried out for approximately 20 minutes.

Pre-forming- cold pressing: Laboratory scale

Calculating from the mould size and the target density of the board the raw material mixture was weighed and spread evenly in the mould. Size of the mould used in the lab was 310 X 250 mm. When about 1kg of the mixture was placed in the mould the height of the mixture spread was 50mm.

The lid of the mould was placed on the mixture and the entire assembly was transferred into the cold press. By cold press we assume room temperature which varied between 15 to 25°C throughout the study. As this step was only used to remove air and to compress the mixture into a mat the temperature variation in different mats was not considered important. Time of pressing in the cold press was approximately 2min. The pressure applied in the cold press was 11kN/ area of un-pressed sample. ($11/30.5 \times 24.5 = 0.0147 \text{kN/cm}^2$). Height of mix in die after cold pressing: 30mm.

Hot Pressing: Laboratory scale

Time of pressing in the hot press: 8min. All mats were pressed at a temperature of 180°C for ten minutes. The average closing time between press contact with the top of the mattress and reaching the target thickness was about 40 seconds. Pressure in hot press: 600psi The dimension of each panel after hot pressing with the use of pressure stops was about 33.5 cm long by 26.5 cm wide by 1.2 cm thick. Height of board made in between Teflon sheets after hot pressing: 12mm. The pressure applied in the hot press was 500psi.

The ultimate objective of the hot pressing operation is to compress the fibre mat and heat it sufficiently for the resin to cure. If the resin is not cured sufficiently the panel may “blow” on press opening or have low mechanical properties. During the hot pressing operation, the resin in the surfaces of a panel cures first as the increase in temperature in this area is most rapid. The resin in the centre of the panel is the last to cure as the temperature in this area takes longest to rise. It is only after the resin in the centre has cured sufficiently that the press can begin to open. When the press opens, load is removed from the panel and there will be residual stresses present perpendicular to the plane (i.e. in the direction that the load was applied).

10 Different types of wood:

Various soft woods and hard woods were used to check whether the different binder system is efficient on the various wood types. It is known that the extractives in the different wood types are different and their acid values are different. This is known to hamper the function of different wood adhesives.

15 Veneer application:

Veneer is a thin surface layer, as of finely grained wood, glued to a base material. It can be glued together in layers to make plywood. In our experiments to add variety and for aesthetic purposes we placed a layer of veneer on the board to see if it could be adhered to the composite substrate and whether any modification was required for its application. In the samples done it was seen that the veneers could be placed either below the mix in the mould that is before the cold pressing stage or above the composite mix before the hot pressing. In both cases the veneer stuck very well to the board. It was also possible to partially coat the board with the veneer.

The composite boards that are made are mostly based on the wood fibre being evenly distributed throughout the board thickness. Layered boards were also made with a layer of veneer, paper, or card board on one side of the board. The additional layer was placed at the base or on the face of the board mixture before the cold pressing. No additional binder was used for the adhesion. The mixture in contact with the layer was sufficient to bring about the adhesion. The adhesion was excellent after being tested for internal bond strength test. The sample is seen to cleave at the centreline and not at the laminate line as seen with all the

other samples with out the laminated layer. A layer of veneer was placed on the board mix with the curved part facing up before placing in the hot press.

Paper layer application:

When in the mould the Board mix was layered with extra powder along the surface and a
5 piece of paper placed on it and then heat cured and excellent adhesion was obtained.

Mouldable boards:

Were able to be made with polyethylene from used milk bottles mixed with the powder/paint binder system.

Wax addition:

10 Mobil wax emulsion from a MDF Mill was added to the board composition. The wax consisted of 65% solids and is usually added at a loading of 0.4% w/w wax solids on oven dry weight of wood chips or fibre in MDF mills. This was added in a 2% composition of the total composite composition. It was assumed that this addition will yield better water resistance properties, but as the boards have good water resistant properties anyway this wax
15 addition was found to have no further advantage.

Acrylic Emulsion:

Two types of Acrylic emulsion polymers were used.

Viscopol 7788, batch #E9251 to this 12 % water was added and then it was used as 20% of the total composite composition.

20 Polyvinyl acetate homopolymer emulsion, Viscopol 6624 from batch # E9512 was also used in the same way by addition of 12 % water and used as 20% of the total composite composition.

Both these solutions gave reasonably good boards but there was concern in them being mixed into a homogeneous mixture. The solutions were seen to be more tacky and had lower
25 spreadability.

Use of MDF Fluff or sander dust:

It is seen that from the furniture industry a lot of wood waste mostly in the form of MDF Fluff or sander dust is generated. Since this contains formaldehyde it cannot be used for example as garden mulch. This type of waste has to be dealt with like chemical waste and has to be disposed off in the land fill according to the current guidelines. In the composite boards made in this study we can make use of this waste source and the subsequent encapsulation by the powder/paint binder system reduce the formaldehyde emissions considerably.

Experiment 1: Laboratory scale

10 Boards made with different compositions – initial work

This was the initial set of boards that were made. There was no fixed preconception as to what the ratio should be of the raw materials. The idea was to arrive at an ideal composite mix of raw materials that will result in good quality boards. We had in mind the WPCP as the binder and the sawdust as the reinforcing fibre. The addition of paint as the secondary binder was just a suggestion at this initial stage. The powder used for this experiment was the TGIC powder. Mixing the sawdust with the binders was done manually for this experimental set and was carried out till reasonable homogeneity of the matrix was achieved. Different compositions that were used are mentioned below in table 6.1. The cold pressing and the hot pressing were done in the standard procedure mentioned in the method of manufacture.

20 We use the coding RPP to refer to “recycled, powder, paint” and the number to show the percentage of powder (shown as WPCP in the table heading, referring to “waste powder coating powder”. RP refers to products made from resin and powder (without the paint).

Table 6.1: Initial raw material mixing ratios

Board Type	Sawdust %	WPCP %	Paint %
RPP25	50	25	25
RPP30	50	30	20
RP25	75	25	--
RP30	70	30	--
RP40	60	40	--

25

Table 6.2: Test Results according to AS/NZS 1859.1:2004 comparing the properties of boards made with different raw material mixing ratios

Sample	d/mm	S_d^{24} (%)	ρ /kg m ⁻³	MC(%)	MOR/MPa	MOE/MPa	I/MPa
RP25	11.66	6.17	647	9	nd	nd	0.49
RP30	11.63	10.83	660	8	10.54	1934	0.27
RP40	12.07	10.6	887	8	12.93	2500	0.17
RPP25	12.42	2.74	735	9	11.80	2182	0.22
RPP30	11.95	2.34	808	9	14.79	2141	0.82

- 5 MRGP = moisture resistant general-purpose boards; HPPB = high performance particleboards; ^a = nominal thickness range; ^b = upper specification limits; ^c = lower specification limits; d = thickness; S_d^{24} = % increase in thickness after 24 h water soak; ρ = density; MC = moisture content; MOR = modulus of rupture; MOE = modulus of elasticity; I = internal bond strength; nd = not done.
- 10 These boards were made as a preliminary exercise. The way, in which the boards were made, were in the primary stage of development. There were variations in the density of these boards as not much planning and calculations had gone into their making. This was more of a feasibility study. The boards with the paint had lower swelling characteristics compared to the boards with no paint. These were the basic compositions under consideration. Physically
- 15 all the boards looked good and there was no particular difficulty in pressing either type. What we notice clearly is that the three point bend strength of the board increases with increase in the powder content where as the internal bond strength has varied results which are related to the quality of mixing and may have bearing on the other properties.

Experiment 2: Laboratory scale

- 20 Boards made with and without the secondary binder

HAA powder was taken from the same batch to make the boards. All the parameters for pressing these boards were similar from the hand mixing of the sawdust with the binders to the hot pressing stage. The only difference was that in one set of boards there was the

secondary binder in addition to the powder to be used as the binder with the sawdust as the filler in board composite. The sawdust used was from the same source. The physical and mechanical test results of the boards are shown in table 6.3. The main aim of this experiment was to ascertain the benefits of using or not using the secondary binder.

5 Table 6.3: Test Results according to AS/NZS 1859.1:2004 comparing the properties of RP30 vs RPP25

Sample	d/mm	S _d ²⁴ (%)	ρ/kg m ⁻³	MC(%)	MOR/MPa	MOE/MPa	I/MPa
All powder boards RP30	12.56	11.5	871	5.7	15.49	2866	0.41
σ	0.20	1.75	70	0.09	1.43	375	0.03
Powder with secondary binder boards RPP25	12.64	12	897	4.2	11.30	2184	0.37
σ	0.41	2.6	84	0.29	1.43	130	0.05

MRGP = moisture resistant general-purpose boards; HPPB = high performance particleboards; ^a = nominal thickness range; ^b = upper specification limits; ^c = lower specification limits; σ = standard deviation; d = thickness; S_d²⁴ = % increase in thickness after 24 h water soak; ρ = density; MC = moisture content; MOR = modulus of rupture; MOE = modulus of elasticity; I = internal bond strength.

15 From the results it can be seen that both the boards show very similar properties. One striking difference seen is that the boards with only the primary binder appear to be much stronger as noted from the modulus of rupture test results, than the ones that have the secondary binder. Durability tests (explained in detail in section 6.6) were performed on both the board types and they were visually inspected. These results, suggest that the boards with the secondary binder have better weathering properties. Weighing both these results it is thought that the durability properties are an important aspect not to be compromised on. For
20 this reason the boards made in the future experiments have the secondary binders in them.

Experiment 3: Laboratory scale

Boards made with different powder types (four chemistries)

The purpose of making boards from the four types of powder coating waste, namely TGIC cured, HAA cured, Hybrid (polyester with epoxy) powders and fines (mixed chemistry), was to establish whether all of the powder types could (or should) be utilised independently, and whether the fines alone were satisfactory binders in imparting the desired board properties.

- 5 All the boards were made with the secondary binder having the RPP25 board composition. The boards were tested and compared with the standard and the results are displayed in table 6.4.

Table 6.4: Test Results with comparison to AS/NZS 1859.1:2004: the effect of powder chemistry on composite board properties.

Sample	d/mm	S_d^{24} (%)	ρ /kg m ⁻³	MC(%)	MOR/MPa	MOE/MPa	I/MPa
Standard	8-12 ^a	25 ^b	n/s	5-13	13 ^c	n/s	0.35 ^c
MRGP	8-12 ^a	18 ^b	n/s	5-13	13 ^c	n/s	0.35 ^c
HPPB	8-12 ^a	12 ^b	n/s	5-13	18 ^c	2700	0.45 ^c
TGIC Board	11.89	13.6	938	2.2	16.0	2235	0.60
σ	0.49	1.5	33	0.33	1.7	181	0.08
Primid Board	12.31	19.7	972	2.3	11.6	1988	0.53
σ	0.18	2.1	30	0.21	0.6	94	0.08
Hybrid Board	12.32	8.2	938	2.4	18.6	2046	0.61
σ	0.22	1.5	29	0.32	1.2	233	0.05
Fines Board	12.42	5.0	918	2.8	12.1	1937	0.44
σ	0.16	0.6	27	0.26	1.0	214	0.07

10

MRGP = moisture resistant general-purpose boards; HPPB = high performance particleboards; ^a = nominal thickness range; ^b = upper specification limits; ^c = lower specification limits; σ = standard deviation; d = thickness; S_d^{24} = % increase in thickness after 24 h water soak; ρ = density; MC = moisture content; MOR = modulus of rupture; 15 MOE = modulus of elasticity; I = internal bond strength.

From the test results it is apparent that the test boards meet most of the specifications for the standard board, moisture resistant general-purpose board and high performance particleboard, and that TGIC and Hybrid boards showed the best properties. The board thickness was close to the target thickness of 12 mm, and the 24 h thickness swell data for

the Hybrid and Fines boards are excellent. This suggests that the boards may be suitable for use in areas where they may be exposed to dampness. Physical examination of the boards revealed that most of the wood particles in the boards were coated with the binder and thus had greatly reduced propensity to absorb water, giving low moisture content for the boards.

5 Overall the bending strength characteristics are good. Though the bending strengths (as represented by MOR) of the Primid and the Fines boards are lower than for the TGIC and Hybrid boards, they are close to the standard and can be said to be satisfactory. The internal bond strengths are all highly satisfactory in relation to the standard specifications. The modulus of elasticity of the boards is lower than expected.

10 The high strength values of Hybrid and TGIC boards compared to HAA boards can be explained with the following reasoning. There are many hydroxyl groups in the Primid structure as it is rightly hydroxyl alkyl amide but on the curing reaction the TGIC – polyester product have more hydroxyl groups. The hybrid powders made with the polyester epoxy component too have many hydroxyl groups as a result of the curing step where as the HAA
15 polyester powder on curing have no hydroxyl groups formed on curing. This explains the reason for the uncured HAA powder being more hygroscopic in nature in comparison to the hybrid and the TGIC uncured powders (these have no hydroxyl groups). The more the hydroxyl groups in a chemical structure we presume that there is more capacity for it to form hydrogen bonds. When these cured powders come in contact with cellulose that has many
20 hydroxyl groups then there are more chances for it to form hydrogen bonds with the cured TGIC powder. This is reflected in the strength characteristics of the boards made with the different powders. We see that the Hybrid and TGIC powder boards have the highest strength followed by the HAA powder boards. See Figures 6.2 and 6.3 for the chemical formulas of these resins.

25 Experiment 4: Laboratory scale

Boards made with varying densities

All boards were made with waste polyester-HAA powders. Boards were made in two compositions as the RP30 and RPP25 boards. Further they were made in four decreasing densities as described as A for the high density to D being the lowest density. The RP30

30 boards were termed as composite 1 and the RPP 25 were called as composite 2.

Table 6.5: Effect of density on board properties

Sample	d/mm	S _d ²⁴ (%)	ρ/kg m ⁻³	MC(%)	MOR/MPa	MOE/MPa	I/MPa
<i>Standard</i>	8-12 ^a	25 ^b	n/s	5-13	13 ^c	n/s	0.35 ^c
<i>MRGP</i>	8-12 ^a	18 ^b	n/s	5-13	13 ^c	n/s	0.35 ^c
<i>HPPB</i>	8-12 ^a	12 ^b	n/s	5-13	18 ^c	2700	0.45 ^c
RP30 A							
RP30 B	12.59	12	800	5.7	11	2866	0.40
RP30 C	12.05	17	670	6.2	6	1555	0.19
RP30 D	11.69	15	450	6.5	0.6	191	0.09
RPP25 A	12.64	12	900	4.2	11	2185	0.37
RPP25 B	12.19	11	750	4.8	9	1560	0.38
RPP25 C	11.35	13	640	5.2	4	740	0.21
RPP25 D	11.63	7	460	5.3	0.8	160	0.09

MRGP = moisture resistant general-purpose boards; HPPB = high performance particleboards; ^a = nominal thickness range; ^b = upper specification limits; ^c = lower specification limits; σ = standard deviation; d = thickness; S_d²⁴ = % increase in thickness after 24 h water soak; ρ = density; MC = moisture content; MOR = modulus of rupture; MOE = modulus of elasticity; I = internal bond strength; Composite 1 with no liquid polymer mix and Composite 2 with liquid polymer mix A, B, C and D denoting various density ranges.

10 The two higher density board sets have met or are close to the standard for general-purpose particle boards for most of the criteria. The 750 to 900 range density boards had similar properties for swell thickness, and the internal bond strengths. Looking at the results of density versus the swell thickness property (Figure 6) there cannot be any particular trend forecasted. It is inferred from physical observations of the samples that the way in which the paint is spread over the area of the sample is important for the water to swell the sample.

15 Since the particle size and resin filler ratio is the same there is not much difference seen in the binding capacity of the resin. Only the way it is dispersed can bring about the difference in the swell property. The higher swell percent of the no paint boards (RP30) having low densities relates, may be to the fact that the wood particles are not well coated with the

20 binder and this may explain the poor durability of these types of boards.

In all boards however it is seen that as the density decreased the strength of the boards were seen to decrease. Further the Composite 2 boards (with liquid polymer mix- the paint) have
5 lower values of strength compared to Composite 1 boards. One reason for this could be that the powder being hygroscopic absorbs water from the paint and on heat application may not bring about complete cure. The presence of paint also implies that there is less amount of powder available as the binder.

Experiment 5: Laboratory scale

10 Boards made with light and dark coloured powders:

When we talk of waste we think of powders being manufactured in different batches and being made of different colours. To get the different colours and shades there have to be added different fillers and pigments. This addition will affect the overall composition of powders. This may have an affect on the binder properties and may affect the properties of
15 the board. To see the effect, of different filler and pigment ratios on the binding capacity of the powders, boards were made with light and dark coloured powders. The powder used for this experiment was the HAA powder in which the resin binder ratios were the same but their filler compositions were different as one was light coloured and the other was dark coloured. The board compositions (RPP25) were the same for the two types of boards made and tested,
20 the only difference being its colour. Both board types had the secondary binder in their composition and were hand mixed till a reasonable homogenous mix was achieved. The sawdust used was from the same source. Boards were made in the standard way and the properties tested and listed in table 6.6.

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Table 6.6: Comparing performance of light coloured powders with dark coloured powders

Sample	d/mm	S _d ²⁴ (%)	ρ/kg m ⁻³	MC(%)	MOR/MPa	MOE/MPa	I/MPa
<i>Standard</i>	8-12 ^a	25 ^b	n/s	5-13	13 ^c	n/s	0.35 ^c
<i>MRGP</i>	8-12 ^a	18 ^b	n/s	5-13	13 ^c	n/s	0.35 ^c
<i>HPPB</i>	8-12 ^a	12 ^b	n/s	5-13	18 ^c	2700	0.45 ^c
Light coloured powder boards	12.64	12	897	4.2	11.30	2184	0.37
σ	0.41	2.6	84	0.29	1.43	130	0.05
Dark coloured powder boards	12.31	19.7	972	2.3	11.55	1988	0.53
σ	0.18	2.1	30	0.21	0.64	94	0.08

MRGP = moisture resistant general-purpose boards; HPPB = high performance particleboards; ^a = nominal thickness range; ^b = upper specification limits; ^c = lower specification limits; σ = standard deviation; d = thickness; S_d²⁴ = % increase in thickness after 24 h water soak; ρ = density; MC = moisture content; MOR = modulus of rupture; MOE = modulus of elasticity; I = internal bond strength.

From the results presented in table 6.6 it can be said that both the board types show not much variation in their board properties. The variation in the internal bond strength test results may be attributed to the not so even mixing that is achieved by the hand mixing process. It can be inferred that the filler composition of the powders does not have any affect on the binder capacity. For future work it can be safely said that boards can be made with any coloured powder coatings waste and it will lead to the manufacture of similar quality boards.

15

Experiment 6: Laboratory scale

Boards with untreated sawdust vs CCA treated sawdust

There is huge amount of preservative treated sawdust that goes to the landfill every day. An effort is made here to see whether this preservative treated sawdust can be used in our board

manufacture. It is of importance to know how the binder efficiency is affected by the preservative treatment of the sawdust. For this purpose two sets of boards were made. One with the normal sawdust, which is made from wood that is not preservative, treated and is environmentally safe where as the other set of boards were made with sawdust derived from wood that is preservative treated. The sawdust particle size was kept similar for both the board sets. The boards were made using the same type of powder (HAA) made with the RPP 25 composition and made in the same way. Both board types had the secondary binder in its composition. The properties were tested and are displayed in table 6.7.

Table 6.7: Comparing performance of boards made with either untreated or preservative treated sawdust

Sample	d/mm	S_d^{24} (%)	ρ /kg m ⁻³	MC(%)	MOR/MPa	MOE/MPa	I/MPa
<i>Standard</i>	8-12 ^a	25 ^b	n/s	5-13	13 ^c	n/s	0.35 ^c
<i>MRGP</i>	8-12 ^a	18 ^b	n/s	5-13	13 ^c	n/s	0.35 ^c
<i>HPPB</i>	8-12 ^a	12 ^b	n/s	5-13	18 ^c	2700	0.45 ^c
Untreated sawdust boards	11.77	8.84	804	3.8	6.6	1822	0.38
σ	0.07	2.95	35.68	0.17	0.26	144	0.05
Treated sawdust boards	11.12	13.57	844	3.68	9.8	1882	0.53
σ	0.22	1.82	44	0.49	1.77	241	0.08

MRGP = moisture resistant general-purpose boards; HPPB = high performance particleboards; ^a = nominal thickness range; ^b = upper specification limits; ^c = lower specification limits; σ = standard deviation; d = thickness; S_d^{24} = % increase in thickness after 24 h water soak; ρ = density; MC = moisture content; MOR = modulus of rupture; MOE = modulus of elasticity; I = internal bond strength.

From the results it can be clearly seen that both the boards have very similar properties. Slight variations are seen, like the swelling characteristics of the treated sawdust boards are higher. As the preservative treated sawdust was derived from construction timber it is assumed to have less lignin content compared to the untreated sawdust, which was derived from a garden centre. The higher amount of lignin in the untreated sawdust may decrease the swelling characteristics. The three-point bend strength and the internal bond strength are lower for the untreated sawdust, which can again be directed to the fact that the lignin

imparts a slight hydrophobic nature to the sawdust. This will hinder the quick spread of the binder during the short hot press stage of board making and a few hydrogen-bonding sites.

5 **Experiment 7: Laboratory scale**

Boards made with different methods employed for the raw material mixing

10 The method of mixing raw materials can affect the properties of composites made from the raw material mixtures. Earlier in the study the raw materials were mixed by hand for board production. Later the mixing of raw materials was carried out in the mixaco mixer. Such mixers can be used for the dry mixing in the powder coating production. Later the extruder was also used and finally the ribbon blender was used. To ascertain the effect of mixing procedure, the composite mixture was mixed by the four methods and boards were made by using the HAA powders. The RPP 25 composition was used for the manufacture. By improving the mixing quality we will be able to get better strength values in future boards.

15 The appearance of boards is directly related to the degree of homogeneity achieved by the mixing technique. The board made from manually mixed composite had patches of binder spread over the surface; the mixaco mixer gave more homogeneity, though the board was still speckled. In the board made from screw extruder mixed composite the binder seemed to have very well coated the sawdust, resulting in a uniform appearance although the extrudate was granular and this was reflected in the appearance of the board. The board made from
20 composite that was mixed in the ribbon blender showed the most homogeneous structure. It resulted in uniform mixing and a consistent appearance from batch to batch.

The effect of the method used for composite mixing on board properties is shown by the data in Table 6.8.

25

Table 6.8: Effect of composite mixing method on board properties

Sample	d/mm	S _d ²⁴ (%)	ρ/kg m ⁻³	MC(%)	MOR/MPa	MOE/MPa	I/MPa
<i>Standard</i>	8-12 ^a	25 ^b	n/s	5-13	13 ^c	n/s	0.35 ^c
<i>MRGP</i>	8-12 ^a	18 ^b	n/s	5-13	13 ^c	n/s	0.35 ^c
<i>HPPB</i>	8-12 ^a	12 ^b	n/s	5-13	18 ^c	2700	0.45 ^c
Manual mixing	11.12	13.6	844	3.7	9.8	1882	0.71
σ	0.22	1.8	44	0.49	1.8	241	0.10
Mixaco mixer	11.14	9.1	832	3.1	10.6	1969	0.70
σ	0.24	1.5	100	0.25	1.9	215	0.06
Screw extruder	11.06	6.1	918	3.5	6.6	2237	0.53
σ	0.22	0.3	27	0.11	0.8	205	0.16
Ribbon blender	11.68	8.2	869	4.0	17.1	2864	1.42
σ	0.40	1.7	71	0.09	3.0	717	0.21

MRGP = moisture resistant general-purpose boards; HPPB = high performance particleboards; ^a = nominal thickness range; ^b = upper specification limits; ^c = lower specification limits; σ = standard deviation; d = thickness; S_d²⁴ = % increase in thickness after 24 h water soak; ρ = density; MC = moisture content; MOR = modulus of rupture; MOE = modulus of elasticity; I = internal bond strength.

The boards made from composite mixed using the twin-screw extruder had the lowest strength (as measured by MOR), possibly because partial curing of the powder occurred as a result of a temperature increase arising from the high shear conditions in the extruder. Manual mixing and the mixaco mixer gave relatively low MOR values of very similar magnitude due to non-uniform resin distribution. Manual mixing also gave comparatively high 24-hour water swell compared to the other boards. The boards made from composite mixed in the ribbon blender had the best overall properties. In conclusion it can be said that the method of mixing is very important to get the best results.

The micrograph of the fracture surface produced in the internal bond test (Figure 3a) shows that the wood component was completely coated with the binders. The sawdust particles were in a flat oriented position due to the compression exerted during board making. Figure

3b shows clearly fractured wood cells, indicating that sawdust particles were fractured rather than being pulled from the matrix.

VARIATIONS

A number of variations have been described above.

- 5 In addition, fire retardant materials, intumescent products, biocides, colourants, etc can be included as additives to the composite material. The disclosed product may possess thermal or sound insulating properties which can be enhanced by varying the formulation, for example the type of bulk filler.

10 This invention may broadly be said to consist in the parts, elements and features referred to or indicated in the specification of the application individually or collectively in any and all combinations of any two or more of the parts, elements or features and where specific integers are mentioned herein which have known equivalents, such equivalents are deemed to be incorporated herein as if individually set forth.

15 Throughout the Statement of Invention, description and claims of this specification the word "comprise" and variations of that word, such as "comprises" and "comprising", are not intended to exclude other additives, components, integers or steps.

ADVANTAGES AND INDUSTRIAL APPLICATION

20 The process and composite materials of this invention enables the conversion of waste plastics coating powder and waste paint to be combined with various fillers to make composite boards of different densities and different properties. Examples include waterproof composite boards made from sawdust and such boards can be used where normal MDF is not suitable. The binder can be manufactured by mixing the powder and paint prior to adding the filler to create a suspension of the powder in the paint, or can be made in situ by mixing the liquid paint with the sawdust and then later adding the powder, or by mixing
25 all three ingredients at the same time to create a slurry, or in some other sequence. In some situations the binder may be mixed from the waste paint and waste powder and sold or delivered to a manufacture of composite materials.

The product and/or method of manufacture can have some of the following advantages:

1. Low cost.
2. Able to use or recycle waste materials.
3. No need to dump waste materials and pay for unnecessary handling.
- 5 4. No toxic materials are added
5. Better able to withstand moisture.
6. Simple manufacturing process (es).
7. Excellent strength characteristics.
8. Better able to be affixed and joined.

10

CLAIMS

1. A liquid or semi-liquid binder system for use in the manufacture of a composite material containing a fibrous or particulate filler and wherein the liquid binder system contains a proportion of a plastics powder coating powder comprising either a cross linkable thermosetting plastics powder or a thermoplastic plastics powder; or a mixture of such powders, in a liquid carrier.
5
2. A liquid binder system as claimed in claim 1, wherein the liquid carrier is selected from liquid paints, water based acrylic paints, solvent based acrylic paints solvent based resin paints, waste paints and water based surfactants.
- 10 3. A composite material containing from 10% to 80% by weight of fibrous or particulate filler and a liquid binder system as claimed in claim 2, wherein the filler is chosen from the group comprising fibrous and particulate waste materials, sawdust, plastic chips and shreds, plant and animal fibres, waste or virgin materials: wool, plastics, rubber, fly ash, glass, sand, polyethylene, polyester fibre, and wood.
- 15 4. A composite material comprising a plurality of particulate or fibrous material encapsulated within a plastics matrix wherein the plastic matrix consists predominately of a thermosetting plastics resin and further includes a percentage of thermoplastic resin.
5. A composite material as claimed in the preceding claim wherein the plastics matrix comprises from 10% to 60% by weight of the composite material.
20
6. A method of manufacturing a composite material selected from the following ingredients:
 - (i) a fibrous or particulate filler and
 - (ii) a liquid binder system containing a proportion of a plastics powder coating powder comprising either a cross linkable thermosetting plastics powder or a thermoplastic plastics powder or a mixture of such powders, in a liquid carrier
25wherein the method includes the following steps of:
 - (a) Mixing the ingredients at low shear to form a mouldable mixture and

(b) moulding and curing the mixture from step (a) to form a solid matrix

7. A method of manufacturing a composite material as claimed in claim 6 further comprising pre-forming or cold pressing the mouldable mixture within a mould to a desired density for a sufficient time, temperature and pressure to allow the composite material to be formed into a desired shaped article.
8. A method for the production of a composite material as claimed in claim 6, wherein the amount of plastics coating powder is from 10%-60% by weight based on the total weight of the composite material, the liquid carrier is from 0.1% - 50% by weight based on the total weight of the composite material and the filler is 10% to 80% by weight based on the total weight of the composite material.
9. A method for the production of a composite material as claimed in claim 7, wherein one or more of the individual ingredients are recycled, scrap or waste material.
10. A method for the production of a composite material as claimed in claim 9, wherein the liquid carrier comprises waste paint.
11. A composite product made from the application of heat and pressure to a mixture containing:
- a) a filler chosen from the group comprising: filler is chosen from the group comprising fibrous, cellulosic and particulate waste materials selected from, comminuted cellulosic packaging materials, sawdust, plastic chips and shreds, plant and animal fibres, waste or virgin materials, wool, plastics, rubber, fly ash, glass, sand, polyethylene, polyester fibre, and wood;
 - b) plastics powder coating powder comprising either a cross linkable thermosetting plastics powder or a thermoplastic plastics powder or a mixture of such powders, which may or may not be suspended in a liquid carrier;
 - c) the liquid carrier chosen from the group comprising: liquid paints, water based acrylic paints, solvent based acrylic paints, solvent based resin paints, waste paints, and water based surfactant mixtures.

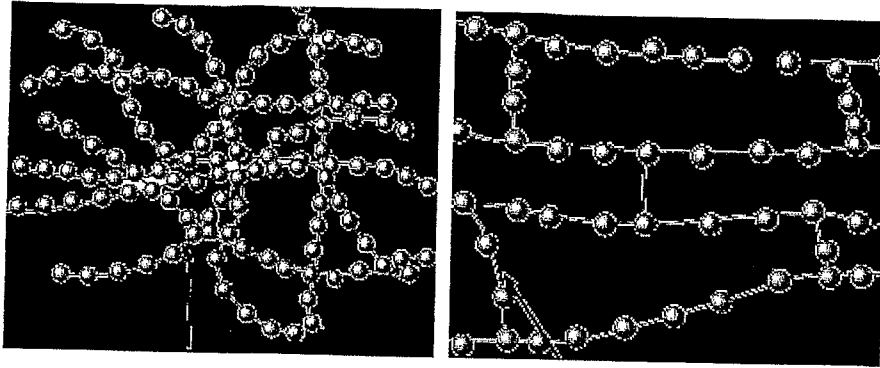


Figure 1a

Figure 1 b

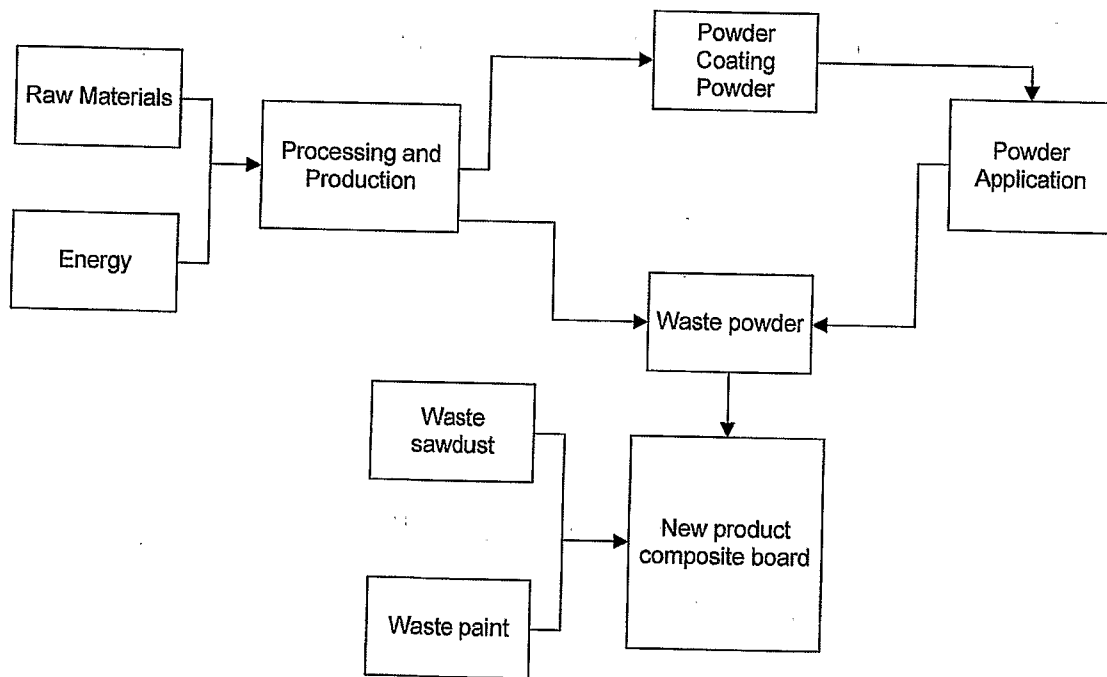


Figure 2

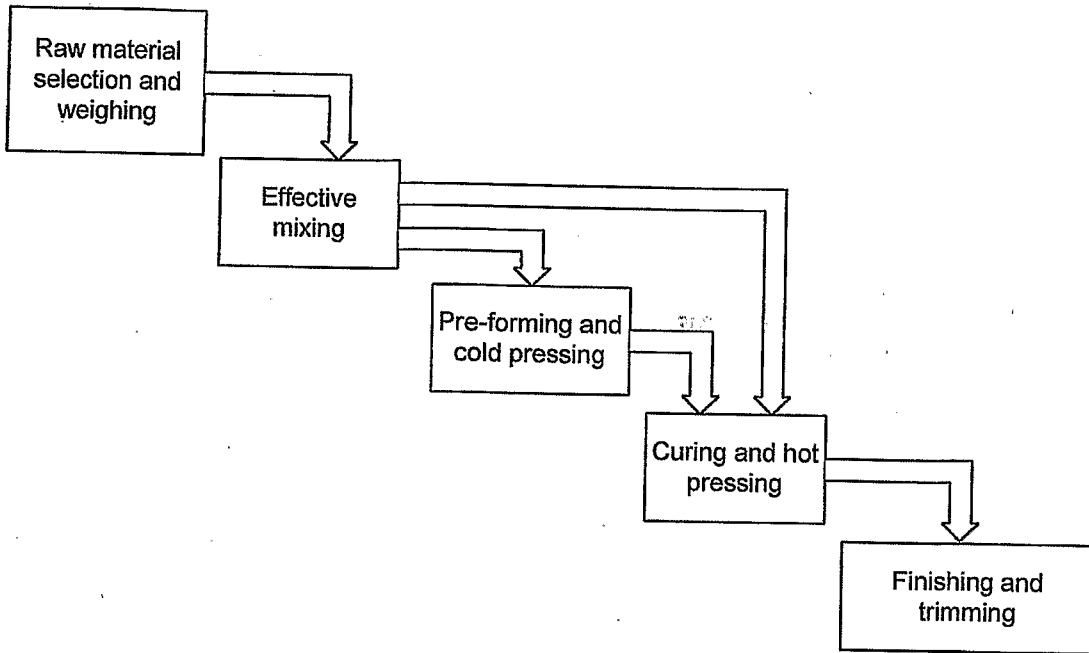


Figure 3

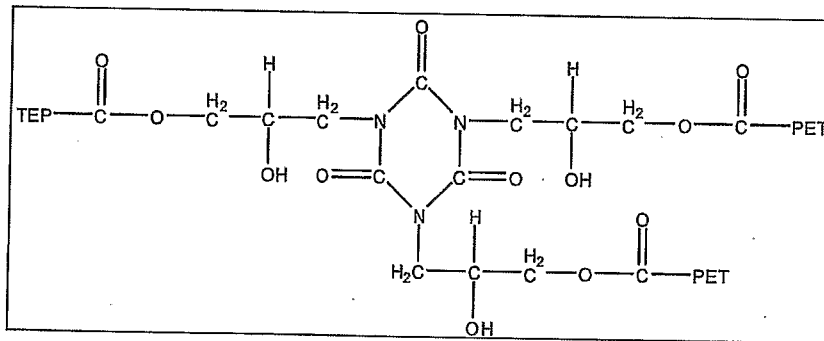


Figure 4

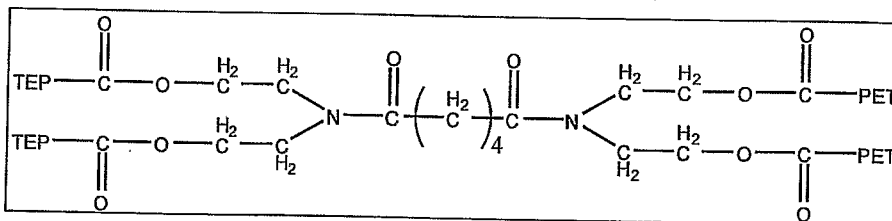


Figure 5

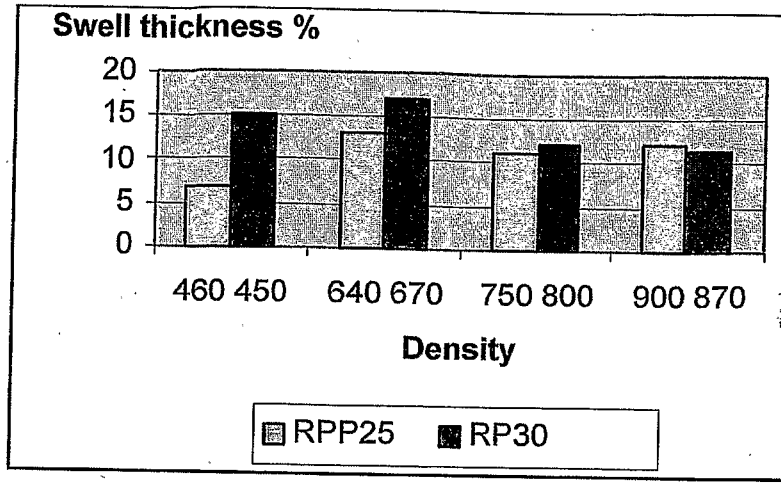


Figure 6

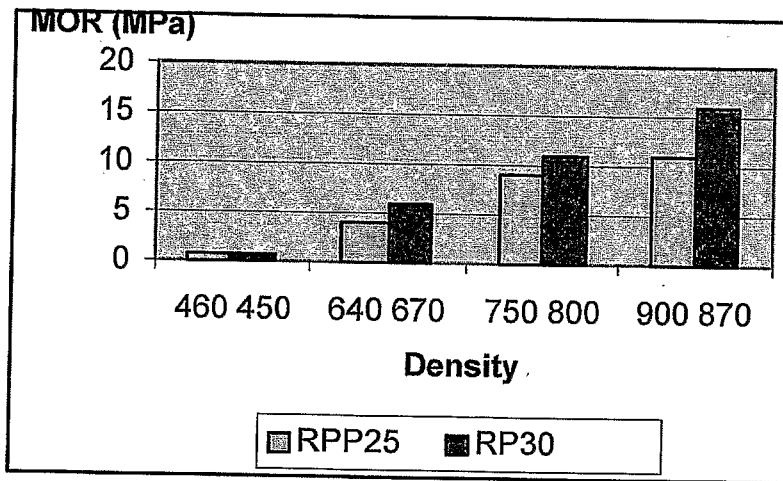


Figure 7

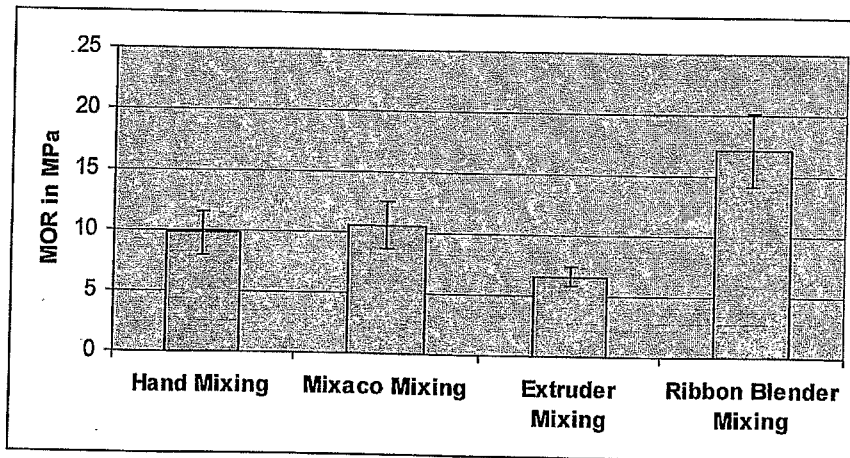


Figure 8

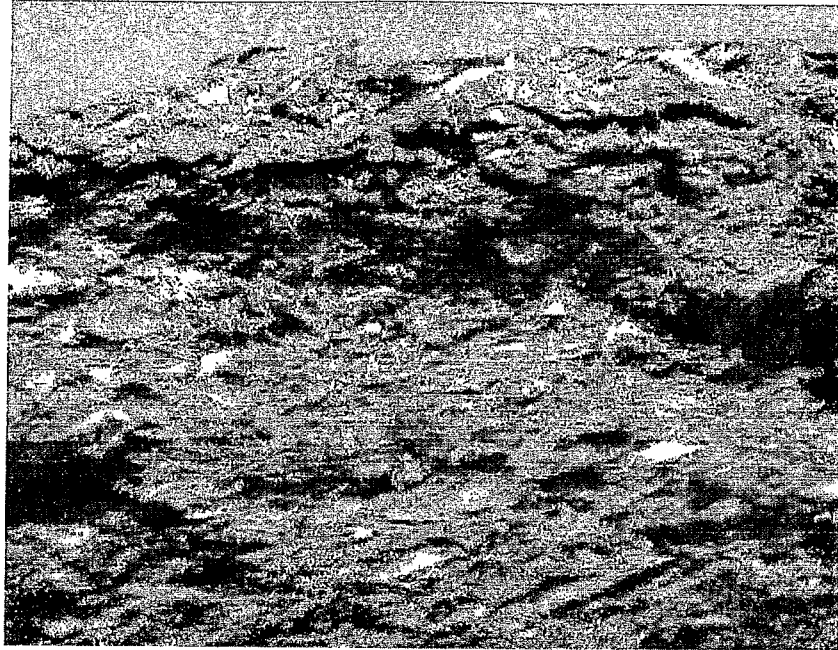


Figure 9

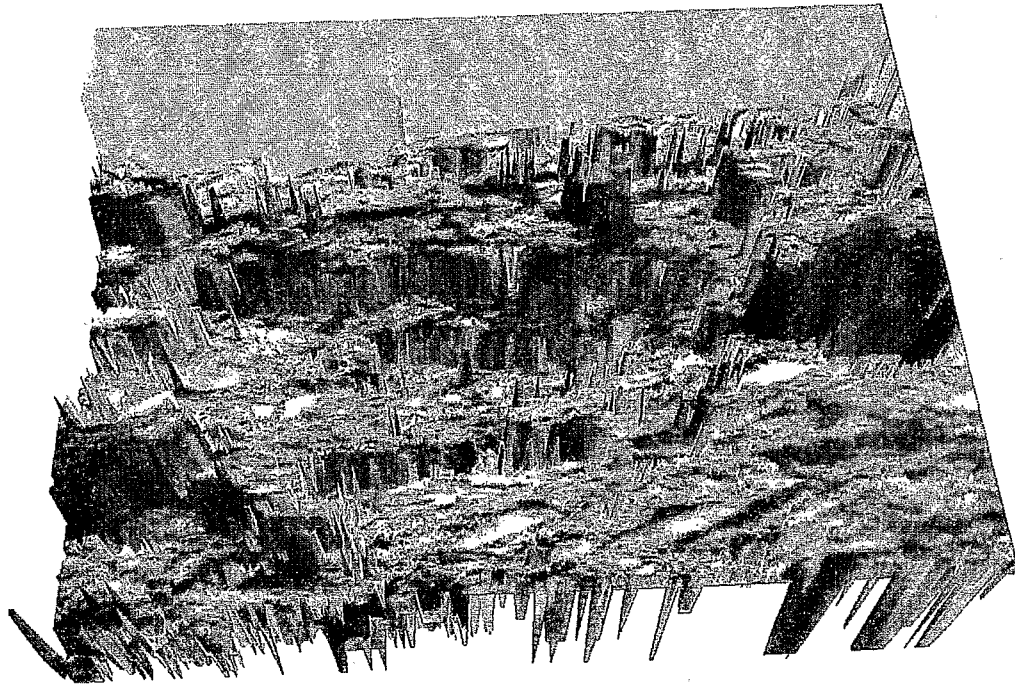


Figure 10



Figure 11a

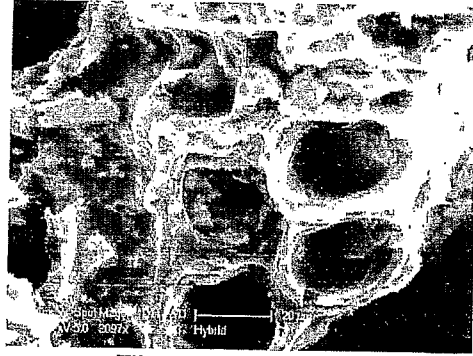


Figure 11b

INTERNATIONAL SEARCH REPORT

International application No.

PCT/NZ2007/000217

A. CLASSIFICATION OF SUBJECT MATTER		
Int. Cl.		
C08J 5/00 (2006.01) C08J 11/00 (2006.01) C09J 201/00 (2006.01)		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols)		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) WPIDS, JAPIO, CAPLUS: & keywords: powder, coat, liquid, solvent, waste, recycle, reuse, composite, & similar terms; composite, thermoplast, thermoset, partic, fibre, matrix, & similar terms		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5852102 A (THYSSEN) 22 December 1998 See the whole document	1-3, 6-11
A	US 6008150 A (THYSSEN et al) 28 December 1999 See the whole document	1-3, 6-11
A	Derwent Abstract Accession No. 1981-04714D JP 55148266 A (NIPPON TOKUSHU TORYO CO LTD) 18 November 1980 See the whole abstract	1-3, 6-11
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C <input checked="" type="checkbox"/> See patent family annex		
* "A"	Special categories of cited documents: document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E"	earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"O"	document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family
"P"	document published prior to the international filing date but later than the priority date claimed	
Date of the actual completion of the international search 11 December 2007		Date of mailing of the international search report 17 DEC 2007
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustalia.gov.au Facsimile No. (02) 6285 3929		Authorized officer ROBYN KNOCK AUSTRALIAN PATENT OFFICE (ISO 9001 Quality Certified Service) Telephone No : (02) 6283 3149

INTERNATIONAL SEARCH REPORT

International application No.

PCT/NZ2007/000217

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE 10129750 A1 (IHD INSTITUT FUR HOLZTECHNOLOGIE DRESDEN GMBH) 9 January 2003 See the whole document	1-3, 6-11
A	US 5215625 A (BURTON) 1 June 1993 See the whole document	1-3, 6-11
X	WO 2005/066244 A2 (THE UNIVERSITY OF SHEFFIELD) 21 July 2005 See the abstract, claims, page 13 lines 10-14, example 4	4, 5
X	GB 2421952 A (UNIVERSITY OF SHEFFIELD) 12 July 2006 See the abstract, page 12 line 24-page 13 line 3, example 2	4,5
X	EP 612812 B1 (IBIDEN CO LTD) 11 July 2001 See the claims, page 7 line 6	4
X	GB 2060490 A (TECHNOCHEMIE GMBH) 7 May 1981 See the abstract, claim 2, example 2	4, 5
X	US 5952435 A (CHIBA et al) 14 September 1999 See the abstract, claims, examples	4, 5

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a)

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

See Supplemental Box

1. As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.
3. As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- No protest accompanied the payment of additional search fees.

Supplemental Box

(To be used when the space in any of Boxes I to IV is not sufficient)

Continuation of Box No: III

This International Application does not comply with the requirements of unity of invention because it does not relate to one invention or to a group of inventions so linked as to form a single general inventive concept.

In assessing whether there is more than one invention claimed, I have given consideration to those features which can be considered to potentially distinguish the claimed combination of features from the prior art. Where different claims have different distinguishing features they define different inventions.

This International Searching Authority has found that there are different inventions as follows:

- Claims 1-3, 6-11 are directed to a binder system and its method of manufacture and use in composites. It is considered that a binder system comprising a proportion of thermosetting and/or thermoplastic powder coating powder and a liquid carrier comprises a first distinguishing feature.
- Claims 4, 5 are directed to a composite material. It is considered that a composite material comprising particulate or fibrous material in a matrix consisting predominantly of thermosetting resin and also including some thermoplastic resin comprises a second distinguishing feature.

PCT Rule 13.2, first sentence, states that unity of invention is only fulfilled when there is a technical relationship among the claimed inventions involving one or more of the same or corresponding special technical features. PCT Rule 13.2, second sentence, defines a special technical feature as a feature which makes a contribution over the prior art.

Each of the abovementioned groups of claims has a different distinguishing feature and they do not share any feature which could satisfy the requirement for being a special technical feature. Because there is no common special technical feature it follows that there is no technical relationship between the identified inventions. Therefore the claims do not satisfy the requirement of unity of invention *a priori*.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/NZ2007/000217

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member					
US	5852102	AU	25234/95	CZ	9603194	EP	0758413
		WO	9530034	ZA	9503558		
US	6008150	AU	41736/96	BR	9509820	CA	2205965
		CZ	9701600	CZ	9804123	DE	4441765
		EP	0793741	HU	77595	PL	320368
		TR	960497	WO	9616218	ZA	9509991
JP	55148266						
DE	10129750						
US	5215625						
WO	2005066244	CA	2552053	EP	1709107	GB	2421953
GB	2421952	CA	2586451	EP	1834173	WO	2006072767
EP	0612812	JP	7033991	JP	2001106921	JP	2001114981
		JP	2006028530	US	5994480	US	6124408
		US	6265498	US	6294621	US	6306980
		US	6451932				
GB	2060490	DE	2941785	FR	2467907		
US	5952435	CA	2184318	JP	9067428		
Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.							
END OF ANNEX							