

Composite Manufacture from Waste Powder Coatings: a Step toward Industrial Ecology

Anuradha C. Abhyankar*, Neil R. Edmonds, Allan J. Easteal
Centre for Advanced Composite Materials, The University of Auckland,
Building 740, Tamaki Campus, Auckland, New Zealand
*aabf001@ec.auckland.ac.nz

Abstract:

Powder coatings are 100% solid, dry powders that are used to coat a wide variety of substrates. They are considered an environmentally friendly coating option with no volatile emissions and minimal wastage. Overspray can be reused for spraying if collected without contamination, but there is a proportion (about 5 to 15%) of powder that has to leave the coating system and is referred to as waste powder coating powder (WPCP). Fines that are not suitable for application are also generated during production, and reworking them into usable powder is a costly and cumbersome process and those materials are not reclaimed. Currently WPCP and fines are disposed of in landfills.

The objective of this work is to make novel products using this waste, thereby eliminating the need for disposal of the powder. Primid powders made by crosslinking polyester with hydroxy alkyl amide (HAA) were used for this study. Composites were made by using WPCP and fines as a primary binder, along with a secondary binder and sawdust as the reinforcing material. Scanning electron microscopy and energy dispersive X ray techniques were used to characterise composite boards formed by compressive moulding of the composites. The composite boards resemble particleboards and were tested for their mechanical properties according to the AS/NZS 1895.1:2004 standard for Reconstituted Wood Based Panels. The test results indicated that the boards met or exceeded relevant standard specifications. As they are constructed from industrial waste materials these particle boards are novel products of sustainable technology, and commercialization of this Industrial Ecological Product is envisioned.

Keywords: Primid powders, powder coatings, waste, fines, particle boards, CCA treated wood waste, physical and mechanical testing of boards, SEM, EDX.

Introduction:

Powder coatings are a dry powder paint technology that is used to coat a wide variety of substrates from metals to specially treated wood. Powder coating powders can be either thermoplastic or thermosetting types. In this study we focus on one type of thermosetting powders (Primid) that are derived from carboxyl functional polyester as the resin and HAA (hydroxy alkyl amide) as the crosslinker. Fillers and pigments are added to make the final powder coating powder. The Primid powders are now the most widely used powders as they are non-toxic and give high quality coatings. The powders cure by condensation reactions in which water is evolved. The powder is usually electro statically sprayed onto an earthed substrate and then heated so that the powder can melt, flow and cure. It is considered an environmentally friendly coating option as there are no volatile emissions during either the coating or the curing step. The wastage is small, in the scene that the overspray can be reused for spraying when coating application is of a single colour. When different colours are sprayed then the amount wasted is higher and is referred to as waste powder coating powder (WPCP). There is always close monitoring of the spray excess powder to prevent it from being contaminated. Eventually there is always some that cannot be reused and is classified as WPCP, and constitutes about 5 to 15% of the total powder sprayed.

During production the particle size of the powder being manufactured is set on the grinding mill, and sieves are employed to monitor the particle size. Air classifiers or cyclones help separate the larger powder particles from the smaller powder particles. The movement of the larger particles is dominated by centrifugal forces and those particles are collected at the bottom or at the periphery of the rotor. The small particles are exposed to a large drag per unit mass and flow along the air stream, finally to be collected at the bag house. This fine powder (fines) is not fit for application and reworking into usable powder is a costly and cumbersome process. When in a particular facility there are different types (colour and chemistry) of powders being manufactured there is a mixed composition of powders in the bag house. It is estimated that about 5% of the powder manufactured is not suitable for use as its particle size is too small.

Currently the fines collected from the bag house are packed in cardboard boxes, moderately heated so as to fuse the powder to form a solid mass and is sent to landfills. Disposal of the fines, wastes raw materials, and incurs costs that are a burden on the manufacturer. Some of those costs are recoverable if the fines can be re-incorporate[1-6] into new manufactured powder. Some attempts have also been made to use the waste powder and fines in insulating blocks[7] and as a slurry[8] to be used as a paint. These techniques are used to a very limited extent for utilising the current annual world WPCP estimate of 60,000 tons (5% of an estimated[9] production of 1,200,000 tons for 2007). When the overspray waste is included the total amount of waste powder is still larger. The aim of this research was to make use of the WPCP and fines as a binder in making composites that are unique and commercially viable materials.

Materials:

Powder: In this study we have considered only thermosetting Primid powders which are made by crosslinking carboxyl terminated polyesters with HAA. This reaction is an esterification reaction[10] between the hydroxy alkyl group and the acid group. HAA reacts with the acid groups at relatively low temperatures (150°C) due to the formation of an intermediate oxazolinium structure. These are condensation reactions in which water is a by-product (Figure 1). Compositions of powders include titanium dioxide, barium sulphate, calcium carbonate, silicates of aluminium, zinc sulphide, magnesium sulphate and calcite along with a host other special purpose agents. These powders are expected to dominate the market in the near future. The polyester powders crosslinked with TGIC (triglycidyl isocyanurate) have recently been tagged as toxic and their use is declining. The Primid thermosetting powders are in the un-crosslinked state. Though the powders used in this study have the same chemistry we have used powders of different colours which in turn have different filler and pigment compositions.

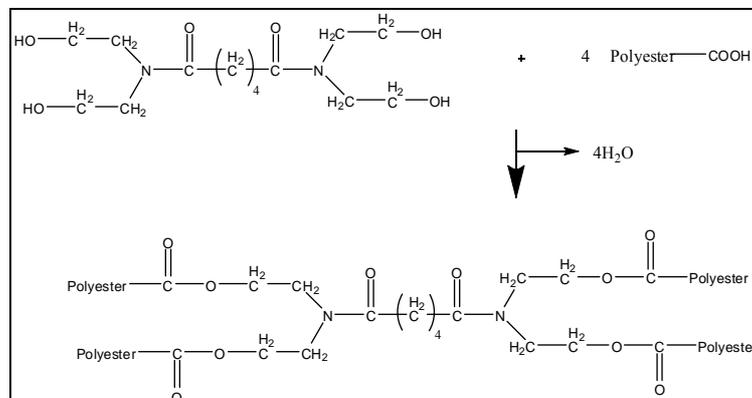


Figure 1: Reaction between hydroxy alkyl group and the acid group from the polyester.

Sawdust: The forest products industry (both primary and secondary manufacturing) is facing a critical problem in disposing of millions of tons of adhesive-coated/chemically treated wood sawdust produced annually. Both untreated (garden safe, not chemically treated) and chemically treated sawdust from *Pinus radiata* was used as a reinforcing component. This was carried out to see if the treatment had any effect on the binding of the composite. The chemically treated sawdust was collected from a saw mill which primarily cut lumber treated to the H3 and H4 standard grades with copper chromium arsenate (CCA). The elemental composition of the elements as measured by X-ray fluorescence is given in Table 1. The particle size of the sawdust was less than 3 mm with about 50% in the range 1 to 2 mm (Figure 2). The sawdust was air dried to maintain moisture content of about 4 to 6% of the dry weight.

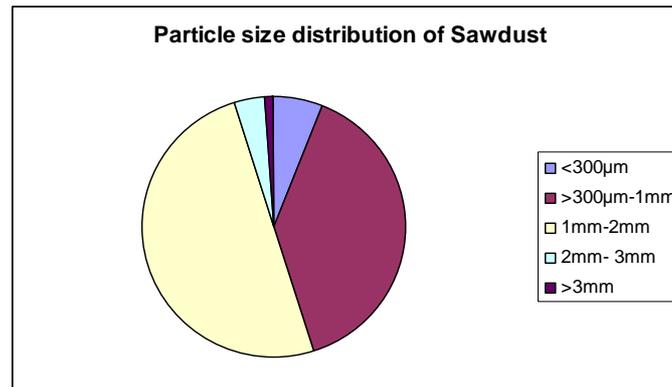


Figure 2: Particle size distribution of sawdust.

Table 1: XRF data for elemental composition of chemically treated sawdust.

Element	Typical Formulation* of Class I (%)	Preservative treated sawdust sample**	Sawdust sample composition (%)
CuO	24.8 (23-25)	0.101	22.8
Cr ₂ O ₃	43.8 (43-47)	0.221	50.0
As ₂ O ₃	31.4 (30-32)	0.120	27.2

*Standards Association of New Zealand, 1992

**Elements (%) detected by XRF of oven dry sawdust sample

The preservative treated sawdust sample was poured into a cup with a thin mylar window and analysed as is. A density of 1.5 g cm⁻³ was used for the calculation. Construction timber that is exposed to weather, above ground has H3 CCA treatment[11] and the H4 treatment is given to timber exposed to weather or that which is used in ground or in fresh water. For above ground construction timber in New Zealand an approximate salt retention of 6 kg m⁻³, (oxide retention of 3.7 kg m⁻³) is specified.

Secondary binder: Originally boards were made with only powder as the binder. After considering other industrial wastes a VOC free industrial liquid polymer waste of known formulation, being 50% solids by weight was selected as a secondary binder. It should be noted that disposal of the waste liquid polymer is of concern in itself. For this study the quality of this polymer waste was kept consistent. The purpose of utilising the liquid polymer waste was to determine whether it enhanced the properties of the composite boards.

Experimental Method:

The composite boards were made with dimensions 310 X 250 mm, by pre-forming in a wooden mould by cold pressing for three minutes under a pressure of 150 kPa using a 100-ton hydraulic press. Then the mould was dismantled and the composite pre-form hot pressed at 160°C in a 50-ton hydraulic press for ten minutes with the use of 3 MPa pressure. A mechanical stop of 12 mm was used to control the thickness of the boards formed. A target

density of 800 to 850 kg m⁻³ was expected. The initial moisture content of the sawdust was about 5% and the composite mixture had a moisture content of about 12%. The composites had a 1:1 weight ratio of sawdust to binder. Three boards of each type were made and cut to form appropriately sized specimens for physical and mechanical property measurements according to the AS/NZS 1895.1:2004 standard for reconstituted wood based panels with the tests from AS/NZS 4266 series. Additional boards that had either one or both binders were tested for the formaldehyde emission characteristics, and were weathered to assess the weathering effect on board structure. The fibre-resin interphase was studied using scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis.

For comparison purposes boards were made (1) with and without the secondary binder; (2) with light and dark coloured powders; (3) with non-treated and preservative treated sawdust.

Results and discussion:

The physical and mechanical test results of the boards are shown in the Tables below for each experiment. The standard specifications are given in the top half of each Table and the experimental results are displayed in the lower half.

Experiment 1: In this experiment boards were made either with powder as the only binder or with powder and the secondary binder.

Primid powder was taken from the same batch, and the sawdust was from the same source for all boards. All the processing parameters for pressing these boards were similar from the hand mixing of the sawdust with the binders to the hot pressing stage. The physical and mechanical test results are shown in Table 2.

Both sets of boards showed very similar properties, except that the boards with only primary binder had substantially larger modulus of rupture and modulus of elasticity than for the boards that included the secondary binder. Both types of board were subjected to weathering by exposure to ambient outdoor conditions for a period of 6 months. Every week the samples were turned to expose all the surfaces. After 6 months weathering the board specimens were visually inspected. In Figures 3a and 3b the left hand images are of control samples that were not weathered; the right hand images are for weathered boards with corresponding composition. Figure 3a shows images for boards with no secondary binder, and Figure 3b images of boards with secondary binder. It is apparent that the boards with secondary binder retained their original form to a greater extent than did boards with primary binder only.

On the grounds that weather resistance is an important property that should not be compromised, in further work the secondary binder was included in composite formulations.

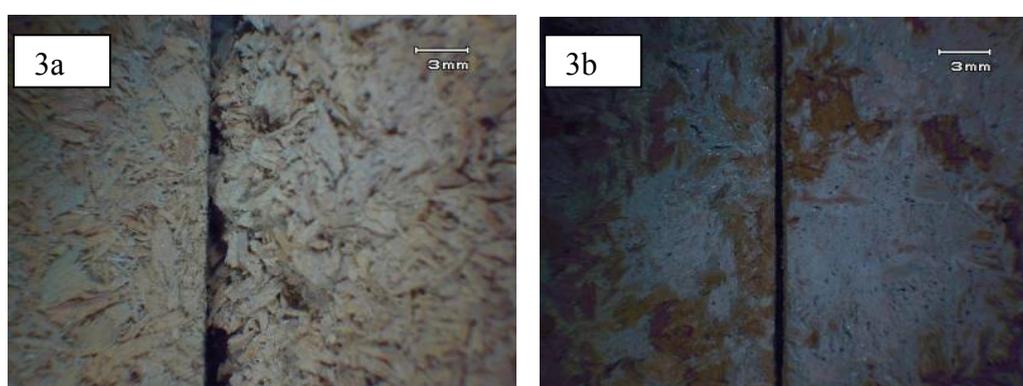


Figure 3. Images of weathered boards and control samples. (a), boards with no secondary binder; (b), boards with secondary binder

Table 2: Test results according to AS/NZS 1859.1:2004 for boards made with and without secondary binder.

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
Without secondary binder	12.56	11.5	871	5.7	15.5	2866	0.41
σ	0.20	1.8	70	0.09	1.4	375	0.03
With secondary binder	12.64	12.0	897	4.2	11.3	2184	0.37
σ	0.41	2.6	84	0.29	1.4	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.

Experiment 2: In this experiment boards were made with light and dark coloured powders to evaluate the effects of filler and pigment ratios on the binding capacity of the powder and hence the properties of boards formed from the powder. The resin:binder ratios were the same for both the powders but their filler compositions were different. The board compositions were the same for the two type of boards, except that one set of boards was made with a white powder coating powder whereas the other set was made with a dark blue powder. Both board types had the secondary binder in their composition. The sawdust used was from the same source. The results of mechanical and other tests are shown in Table 3.

It is apparent that the light and dark coloured powders gave boards with very similar properties, except for water swell which was larger for the boards made from the dark powder. It can be inferred that the filler composition of the powders has very little effect on the binder capacity.

Table 3: Test results according to AS/NZS 1859.1:2004 for boards made from light and 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	12.64	12.0	897	4.2	11.3	2184	0.37
σ	0.41	2.6	84	0.29	1.4	130	0.05
Dark coloured powder	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

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.

Experiment 3: Boards were made with untreated and preservative treated sawdust, to determine whether binder efficiency is affected by the preservative treatment of the sawdust. The sawdust particle size was kept similar for both sets of boards. The boards were made using the same type of powder and made in the same way. Both board types included the secondary binder. The properties of the boards are shown in Table 4.

The two types of boards had very similar properties. The differences in properties (eg water swell) may be attributable to the higher lignin content of untreated sawdust.

Table 4: Test Results according to AS/NZS 1859.1:2004 for boards made with either untreated or 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	11.77	8.8	804	3.8	6.6	1822	0.38
σ	0.07	3.0	36	0.17	0.3	144	0.05
Treated sawdust ds	11.12	13.6	844	3.7	9.8	1882	0.53
σ	0.22	1.8	44	0.49	1.8	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_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.

Formaldehyde Emission Tests:

Recently there has been increasing interest in development of wood based panels with very low formaldehyde release comparable to that present in the atmosphere. Strict standards are in force to regulate the amount of formaldehyde present in building materials. Manufacturing processes and climatic factors influence the evolution of formaldehyde from particle boards. In traditional particle board making it was thought that the higher the formaldehyde content in the resin; the better the strength of the particle board. It has been reported by Aydin et al[12] that as the moisture content of wood veneers increases the formaldehyde emission is lower. On the other hand, lowest mechanical properties were found for plywood panels manufactured from veneers conditioned to 16-18% moisture content. With this in mind both the board types, namely Composite 1 boards without secondary binder, and Composite 2 boards with secondary binder were tested for formaldehyde emission according to the Japanese Industrial Standard JIS5908, by the desiccator method. Composite 1 board gave 0.05 mg l⁻¹ formaldehyde emission and Composite 2 gave 0.00 mg l⁻¹, thereby meeting the standard for the F**** grade. In our boards since there is no formaldehyde in the binder system there is no formaldehyde emission, which is an advantage for use in interior construction work for housing and offices.

SEM and EDX Results:

Fracture surfaces were observed by SEM to study the fibre/resin interphase. The internal bond strength test showed (Figure 4) very good coverage of the sawdust particles with the primary binder only (Figure 4a) or both binders (Figure 4b). During the hot pressing stage the powder melts and flows around the sawdust particles. With further heating under pressure it cures while encapsulating the wood particles. The wood fibres are flattened due to the compressive moulding step. There is little difference between the boards made with a single binder and with both binders.

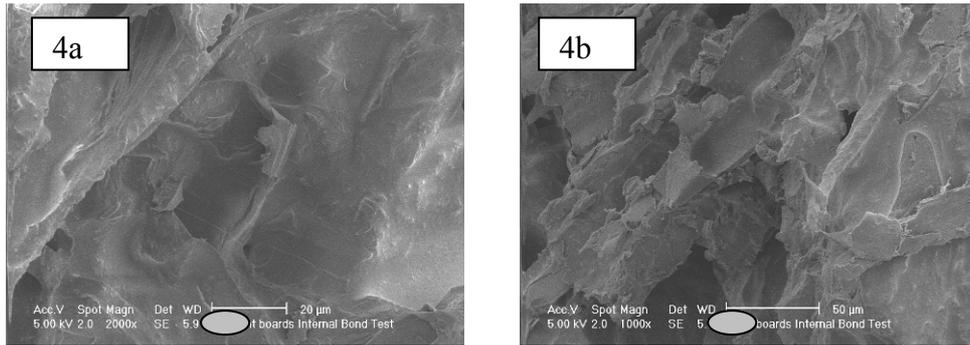


Figure 4a: Scanning electron micrographs of fracture surfaces of boards with (a) only primary binder; (b) primary and secondary binders

When three point bend tested sample surfaces were analysed it was observed that the major rupture occurs in the binder phase and the crack propagates around the sawdust particles (Figure 5). Again in these boards not much difference can be seen in the fibre/resin interphase. Furthermore it is clear from Figure 5a that the fibres are released from the matrix during fracture, indicating a weak bond between the two phases. There are cracks seen in the other composite but they are less prominent. This suggests that there is stronger interaction between the wood particles and binders when both binders are present than in the case of primary binder alone. At the fracture surface it can be seen that the binder coated sawdust particles are ruptured. This is the result of stress transfer from the weaker matrix to the stronger wood fibre. Since the sawdust particles are still coated with binders we can infer that there is good adhesion between the phases.

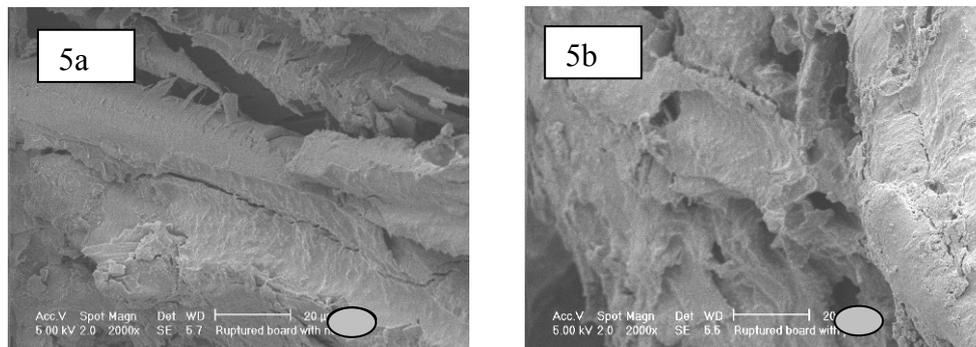


Figure 5 Scanning electron micrographs of MOR test fracture surfaces of boards with (a) primary binder only; (b) both primary and secondary binders.

On further detailed analysis it is apparent that wood particles rupture, leaving open cell structures exposed, rather than being pulled out of the matrix, indicating a well-formed composite. In Figure 6c ruptured wood fibres are clearly visible. On a suitable ruptured wood cell surface EDX was performed (Figure 6d) and compared with that for sawdust (Figure 6a) and powder (Figure 6b). For the sawdust no metallic elements were detected, whereas in the powder sample titanium, sulphur, aluminium, magnesium, zinc and calcium were found. EDX analysis of the marked area (Figure 6c) of the wood cell wall shows the presence of titanium, sulphur, aluminium, zinc and calcium from the powder, confirming that the powder has entered the wood cell walls as the result of good penetration of the binder into the wood cells as a result of elevated temperature and pressure. As reported by Glindl et al[13] penetration of the binder into the fine microstructure of wood increases the area of contact between the wood and the binder resulting in most durable structural bonds.

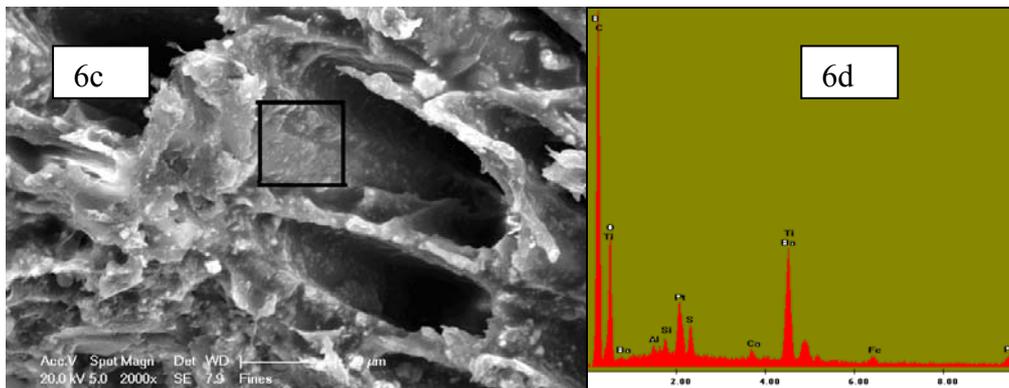
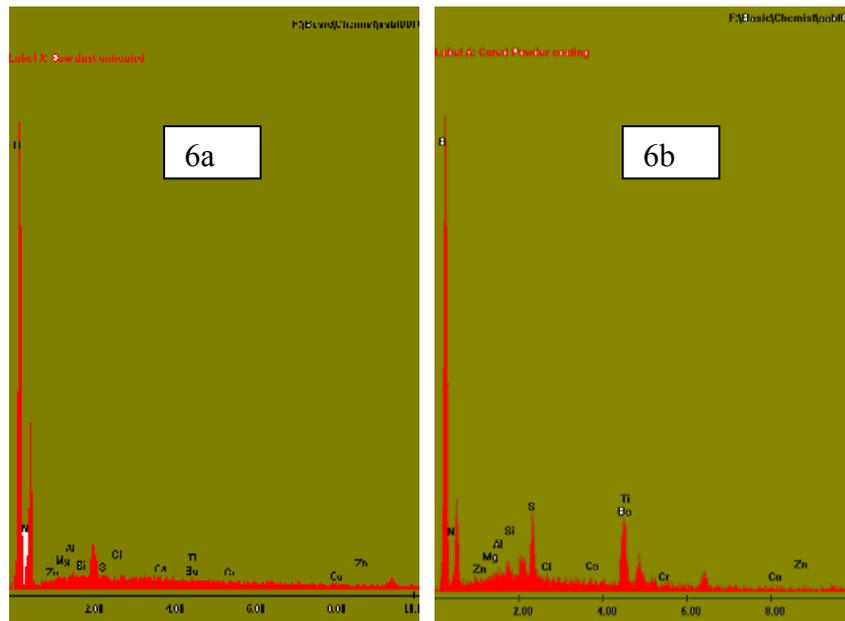


Figure 6: EDX analysis of (a) sawdust; (b) Primid powder. Figure 6c is a scanning electron micrograph of exposed wood cells on a composite fracture surface. Figure 6d is an EDX analysis of the marked area in Figure 6c.

Conclusions:

The aim of effective value addition to waste powder coatings, in making them into particleboards has been achieved. The particleboards are made wholly from waste materials, and if these boards were made on a commercial scale the environmental burden from disposal of the wastes could be greatly reduced. Composites can be made with only powder coating powder as the binder but the addition of a secondary binder (VOC free industrial liquid polymer waste) improves weathering characteristics. All Primid waste can be used, and sawdust from both untreated and preservative treated wood can be utilised. The sawdust particles are sufficiently well coated with binder and the risk of exposure to CCA treated wood particles is minimal.

The boards made in this study are formaldehyde free, and board properties meet the relevant standard specifications so we expect that they will have a place in future commercial usage[14].

References:

- [1] K. Blatter, D. Montgomery, H. Bolm, *Method for processing coating powder waste and coating powders thus obtained*, US Patent 6433030.
- [2] K. Blatter, D. Montgomery, H. Bolm, *Continuous method for reusing coating powder waste and coating powders thus obtained*, US Patent 6500385.
- [3] R. Graewe, *Process for recycling powder coating waste*, US Patent 6881762.
- [4] R. Graewe, *Process for recycling of powder coating waste*, US Patent 6428843.
- [5] R. Graewe, *Process for recycling of powder coating waste*, US Patent 6635207.
- [6] I. Kilner, *Process for recycling powder coating fines*, GB Patent 6793163.
- [7] B. Murray, A Novel Approach to the re-use of Powder Coating Fines, in: *Paper presented at Powder Coating 2002*, Indianapolis, 2002.
- [8] Anon, Aqueous Powder Systems, *Finishing Industry*, 5 (1982) pp. 21-22.
- [9] T. Wright, Chinacoat 2006, *Coatings World*, December, 2006, pp. 26.
- [10] P. G. d. Lange, *Powder Coatings Chemistry and Technology*, Vincentz Network, Hannover, 2004.
- [11] D. Read, *Report on Copper, Chromium and Arsenic (CCA) Treated Timber*, Environmental Risk Management Authority, NZ, 2003.
- [12] I. Aydin, G. Colakoglu, S. Colak, C. Demirkir, Effects of moisture content on formaldehyde emission and mechanical properties of plywood, *Building and Environment* 41(10) (2006) pp.1311-1316.
- [13] W. Gindl, E. Dessipri, R. Wimmer, Using UV-Microscopy to Study Diffusion of Melamine-Urea-Formaldehyde Resin in Cell Walls of Spruce Wood, *Holzforschung*, 56 (1) (2002) 103-107.
- [14] G. N. Squire, A. Abhyankar, K. J. Watson, *Product and method of manufacture*, NZ Patent Application 549170.