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UTILIZATION OF SOME AGRICULTURAL WASTES

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UTILIZATION OF SOME AGRICULTURAL WASTES

THE possibilities of the utilization of agricultural wastes has engaged the attention of workers in various parts of the world and in recent years practical uses for some of these residues have been developed. This booklet deals with the work carried out on the subject by the author during the past several years.

These materials may be used as animal and poultry litters, animal feeds, mulches, fertilisers and ingredients of insecticides and pesticides, etc., in the field of agriculture only. The materials may form the source of chemicals obtained by destructive distillation (tars); by acid hydrolysis (furfural) or by extraction (tannins, etc.). Other applications based on their physical properties may include : thermal insulation, as cleaning and polishing agents, filler for soaps, bricks, ceramics, etc., or in the adhesives and plastics industry, etc.

The main applications studied were: (i) insulation wools, (ii) insulation and hard boards, (iii) adhesives, (iv) furfural production, and (v) filler for adhesives and plastics.

The wastes examined included plant-stems, husk of grains, nutshells and seed cakes. The last item will be dealt with in a separate publication. This publication deals with (i) plant stems, e.g., tapioca stems, bagasse, water hyacinth, (ii) other plant residues like linseed straw, tea waste, spent tea leaves, etc., (iii) arecanut husk, and (iv) nutshells like coconut shell and walnut shell.

Chemical Composition of the Wastes Studied

The chemical composition of the wastes studied is given in Table 1.

TABLE 1. CHEMICAL COMPOSITION OF DIFFERENT WASTE MATERIALS

Material	Moisture per cent.	Ether extract per cent.	Alcohol benzene extract per cent.	Hot water extract per cent.	Pentosans per cent.	Lignin per cent.	Cellulose per cent.	Ash per cent.
Arecanut husk	8·8	0·93	2·04	17·12	28·17	27·04	64·79	4·75
Coconut shells	6·76	0·169	2·01	1·83	31·93	30·15	51·6	1·32
Tapioca stems	7·46	1·78	2·66	16·67	19·15	40·23	48·54	4·78
Spent tea leaves	5·72	4·60	8·40	11·78	15·60	40·25	48·20	4·53

Insulation Wools

Wood wools find application in various industries for thermal insulation, acoustic correction, etc. During the war, substitutes for materials like palco wool were in demand by the Defence and other Services. Experiments at the Institute indicated that a satisfactory wool could be produced by beating the air-dry husk of arecanut. The wool so produced as well as the pods of *Prosopis stephaniana*, as can be seen from Table 2, had satisfactory properties.

TABLE 2. THERMAL CONDUCTIVITY OF SOME INDIGENOUS INSULATION WOOLS

Material	Density of packing gm/cm.^{3}	Moisture content per cent.	Mean temp. ^{0}C.	Thermal conductivity Kcal/mh^{0}C.
Arecanut husk wool ...	0·08	9·9	17·00	0·0394
Pods of *Prosopis stephaniana*	0·08	13·5	18·00	0·0410
Defibrated teak bark ...	0·08	8·3	16·83	0·4468
Palco wool ..	0·08	6·2	16·43	0·0431
Granulated cork ...	0·09	20·0	20.00	0·0350

As can be seen, arecanut husk wool is not inferior to foreign materials. The improvement of the durability and other properties of the wool are under investigation.

Insulation Boards

Insulation boards have been prepared by the defibration process from arecanut husk and tapioca stems. The material was soaked in cold water for the optimum period with arecanut husk and for 8 days with tapioca stems and then defibrated in a laboratory Asplund defibrator for a suitable time at the appropriate temperature, then washed and the pulp mixed with the necessary quantity of water, and formed into a felt like sheet (9 in. × 9 in.) in a laboratory board forming machine. These sheets were placed between the hot platens of a hydraulic hot press with wooden battens of thickness required on all sides, and dried without pressure at 100°-150°C in 2-3 hours. When desired the boards can be rendered resistant to fungi and termites by the addition of sodium pentachlorophenate to the pulp. The properties of insulation boards so made from arecanut husk and tapioca stems are given in Table 3.

TABLE 3. PROPERTIES OF INSULATION BOARDS

Material (board)	Thickness cm.	Sp. gr.	Moisture per cent.	Mean temp. °C.	Thermal conductivity Kcal/mh°C.	Flexural strength lb./sq. in.
Arecanut husk	—	0·517	—	22·25	0·0735	
Tapioca stem	1·692	0·292	6.49	27·89	0·0593	237·5
-do-	1·634	0·291	6·19	28·79 29·35	0·0475 0·0477	186·7
-do-	3·141	0·172	8·67	25·60	0·0425	—
-do-	3·189	0·161	9·02	25·23 25·48	0·0393 0·0382	—
Celotex	—	0·328	—	28·42	0·0393	—
Slab cork	—	0·181	—	20·95	0·0410	—

Table 4. Sound Absorption and Flow Resistance Properties of Tapioca Stem Fibre Boards

Thickness (cm.)	Sp. gr.	Moisture content (per cent.)	Permeability (cm.^{3} sec. g^{-1})	Standard pressure drop ratio for unit thickness (cm^2.)	Porosity (per cent.)	Flow resistance (cm.$^{-3}$ sec. g^{-1})	Sound absorption coefficient at 512 cycles per second
1.692	0.202	6.46	2.049×10^{-4} 1.824×10^{-4} 1.448×10^{-4} 1.998×10^{-4}	1.585×10^{-7} 1.411×10^{-7} 1.121×10^{-7} 1.555×10^{-7}	80.13	2.539×10^{5} 2.749×10^{5} 3.268×10^{5} 2.370×10^{5}	— — — —
1.634	0.291	6.19	2.936×10^{-4} 2.518×10^{-4} 2.411×10^{-4} 2.717×10^{-4}	2.206×10^{-7} 1.890×10^{-7} 1.812×10^{-7} 2.041×10^{-7}	80.21	1.666×10^{5} 1.830×10^{5} 2.031×10^{5} 1.799×10^{5}	— — — —
3.141	0.172	8.67	4.021×10^{-4} 3.999×10^{-4}	5.785×10^{-7} 5.756×10^{-7}	88.22	6.983×10^{4} 7.015×10^{4}	— —
3.189	0.161	9.02	4.132×10^{-4} 4.205×10^{-4}	6.029×10^{-7} 6.211×10^{-7}	88.98	6.458×10^{4} 6.316×10^{4}	31%
0.350	1.150	6.14	1.160×16^{-7} 1.204×10^{-7}	1.860×10^{-11} 1.930×10^{-11}	20.55	5.043×10^{8} 4.858×10^{8}	— —

The sound absorption and flow resistance properties of the boards are given in Table 4.

The boards compare favourably with similar commercial products with regard to appearance and thermal conductivity. The sound absorption and flow resistance properties are also satisfactory.

With world shortage of building materials of all kinds, insulation and hard boards derived from various ligno-cellulosic wastes have assumed great importance. The production of fibre boards in Sweden had a five-fold increase in 1953 as compared to 1938 Hard board output was proportionately higher than the insulation boards in 1951 as compared to 1938. In recent years, hard boards have gained importance at the expense of other wood products, especially plywood. The increase in price during 1937-53 of hard boards is less than that of other products. This is attributed to the fact that the industry is new and developing. Thus with technological improvements, costs are bound to go down. Raw material costs also form a smaller proportion of manufacturing costs. While for the production of fibre boards wet processes are used, there are several dry processes also. While plants for the production of various types of building boards are in operation in Europe and America, Japan is the only country in Asia which has a board industry. Nevertheless, the importance of these materials for the balanced timber economy of the country has induced studies in this field. Various types of building boards have been developed from agricultural and forest wastes. Both wet and dry processes have been used.

Both arecanut husk and tapioca stems have been found to form satisfactory materials for the production of hard boards by the Asplund process. Pulp yields with tapioca stems varied from 62 to 88 per cent. depending on the conditions of preparation. The strength and other properties of the boards were as given in Table 5.

In later experiments, pretreatment of arecanut husk with sodium hydroxide was tried. The beaten husk was boiled in a solution of 0·5 to 2 per cent. sodium hydroxide and kept at about 100° C. for one hour, taking care to replace the water evaporated, by addition of fresh water

TABLE 5. PROPERTIES OF BOARDS MADE OUT OF ARECANUT HUSK AND TAPIOCA STEMS

Material	Pressing conditions			Moisture (per cent.)	Sp. gr.	Modulus of rupture (lb./sq. in.)	Tensile strength (lb./sq. in.)	Water-absorption 24 hrs. (per cent.)	Swelling (per cent.)
	Pressure (lb./sq. in.)	Temp. (°C.)	Time (mins.)						
Tapioca stem	200	150	60	6.82	0.76	3937	2660	65.32	54.79
do.	500	150	60	6.95	1.02	6115	3290	44.53	52.78
do.	800	150	60	6.76	1.05	6205	3450	43.67	50.72
do.	2250	150	60	6.05	1.13	9210	5405	39.32	45.72
do.	2000	150	60	6.14	1.15	9535	...	26.23	15.36
do. (tempered)	,,	,,	,,	6.06	1.16	11537	...	17.23	7.86
Arecanut husk					0.95	13700	2400		
do.	750	150	30	5.5	1.19	5150	2600	22.7	15.0
do.	1000	150	30	4.5	1.23	5600	3000	15.5	9.1

at intervals. At the end of the treatment the liquor was allowed to drain and the material washed with water till the wash water was clear. It was then defibrated for 5 to 7 minutes at room temperature in the presence of water and the pulp again washed. It was then formed into a sheet and pressed. The results are given in Table 5.

The strength and moisture absorption properties of these boards were found to be further improved by treatment of the pulp with p.f. resin but such treatment increased the cost.

Thermal Plasticisation

Many processes of fibre board manufacture are based on the thermal plasticisation of ligno-cellulosic materials and activation of the lignin present in them, viz., the Masonite process, the Asplund process, etc. It is held that a partial hydrolysis takes place, the hemicelluloses are largely hydrolysed, the lignin-carbohydrate bond is ruptured, the lignin is activated and various resin forming substances which are produced condense to form plastics Recently, Runkel (1951) has developed a process in which the wood is heated with very little water in a closed container without injurious space and simultaneously pressed into boards. At the Institute various agricultural wastes like tapioca stems, coconut shells and arecanut husk have been subjected to a thermal treatment in the presence of water (vide Narayanamurti *et. al.*) in an autoclave at above 190°C. and then pressed to boards. The equipment is simpler than that used by Runkel. The properties of some of the boards so prepared are given in Table 6.

TABLE 6. PROPERTIES OF BOARDS MADE BY THERMAL PLASTICISATION

Material of board	Density (gm./cm^3.)	Moisture (per cent.)	Modulus of rupture (lb./sq. in.)	Water absorption (per cent.)
Tapioca stem (dust)	1.32	4.35	4046	4.69
do.	1.37	5.68	6487	4.56
Coconut shells (sawdust filler)	1.44	5.62	6385	3.52
Coconut shells (bamboo dust filler)	1.30	5.39	5625	3.50
Arecanut husk	1.39	7.0	4750	10.5

Dry Processes

Several dry processes were also tried, viz., the paraformaldehyde process, and use of chemicals other than synthetic resins.

The production of building boards from barks containing tannins by the addition of small amounts of paraformaldehyde has been studied by Bryant (1951) and Narayanamurti *et al.* in this country. Catechol or phlobotannins will react with formaldehyde to form resin. The boards were usually made as follows : The air-dry material was powdered in a disintegrator to pass through 60 mesh sieve and then mixed with sawdust (40 mesh, 8 per cent. moisture content) The amount of paraformaldehyde to be added could be roughly estimated by the addition of formalin to the aqueous extract of the powder Usually 15 gm. of the powdered material were extracted with boiling distilled water for 3 hours and the extract made up to 2 l. Dilute solutions of formaldehyde were added to aliquots of the acidified extracts and the precipitated 'tannin formaldehyde resins' were dried and weighed. It was found that quantities equivalent to 1.0 per cent paraformaldehyde were sufficient to precipitate tannins in the case of arecanut husk.

The powdered material with the requisite quantity of paraformaldehyde and water were mixed well in an edge runner, put in a mould and pressed at a suitable pressure and temperature, (Table 7). Best results were obtained when the moisture content of the mixture was about 14 per cent.

Othmer and Riccardi (1950) claim to have developed a process in which some cheap chemicals instead of resin binders are said to be used for making boards from sawdust and similar materials. Some years back during the course of other studies at the Institute, it was noticed that fungus-attacked-wood could be moulded under heat and pressure into water resistant boards of satisfactory strength. Based on this work several chemicals, e g , polybasic organic acids and phenolic bodies etc. are added to sawdust and other ligno-cellulosic wastes which could then be dry pressed at about 800 lb./sq. in. at 150°C to satisfactory boards* The properties of these are given in Table 8.

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* Vide Narayanamurti and J. George, Indian Patent No. 47411

Table 7. Properties of Boards Made by Dry Process

Material	Aldehyde (per cent.)	Pressing conditions		Sp. gr.	Moisture (per cent.)	Modulus of rupture (lb./sq. in.)	Water absorption 24 hrs. (per cent.)	Swelling 24 hrs. (per cent.)
		Pressure (lb / sq. in.)	Temp. (°C.)					
Spent tea leaves	Paraformaldehyde 1.0	750	140	1.42	12.0	2925	25.6	11.3
Spent tea leaves and sawdust (1:1)	1.0	750	140	1.45	12.9	3150	38.1	21.0
do.	0.5	750	140	1.40	12.7	3235	27.9	19.0
Spent tea leaves	Furfural 7.5	1200	150	1.46	9.6	2368	8.5	14.2
Spent tea leaves and sawdust (3:1)	7.5	1500	150	1.41	12.7	3772	13.5	10.7
Arecanut husk and sawdust (1:1)	Paraformaldehyde 1.5	2000	150	1.33	8.4	4540	25.6	12.7
do.	1.5	1000	150	1.27	7.6	3840	30.1	14.8
do.	Furfural 5.0	1000	150	1.32	7.1	3970	24.8	10.5

TABLE 7. (*Continued*)

Material	Aldehyde (per cent.)	Pressing conditions		Sp. gr.	Moisture (per cent.)	Modulus of rupture (lb./sq in.)	Water absorption 24 hrs. (per cent.)	Swelling 24 hrs. (per cent.)
		Pressure (lb./sq. in.)	Temp. (°C.)					
Tea waste	Furfural 5 CaO 6.5 Na_2CO_3 0.35 CNSL 0.35	—	—	1.40	5.5	2580	17.2	—
Tea waste and sawdust (1:1)	do.	—	—	1.31	11.7	4110	5.7	—
do (9:1)	do.	—	—	1.39	7.7	4034	7.4	—

TABLE 8. PROPERTIES OF BOARDS MADE BY DRY PROCESS, USING CHEAP CHEMICALS*

Material of board	Activator	Phenol	Moisture (per cent.)	Density (gm./cm.^{3})	Modulus of rupture (lb./sq. in.)	Impact test	Water absorption (per cent.)	Swelling (per cent.)
Tapioca stem	Tamarind	Wood tar phenol	—	1.117	4563	—	18.7	8.01
Bagasse	,,	Cresol						
Linseed	,,	,,						
Jute stalks	,,	,,		Satisfactory boards were formed				
Corn cobs	,,	,,						
Spent tea leaves and sawdust (1:1)				1.21	3258		22.2	7.6
Coconut shells		—		1.29	5262		12.2	9.3
do.	Tartaric acid	Phenol		1.30	5004		17.4	9.37
Arecanut husk	Oxalic acid	Wood Tar phenol		1.14	3500		17.7	5.4

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* Process patented

TABLE 9. RESULTS OF TESTS ON PLYWOOD PREPARED WITH COCONUT SHELL ADHESIVE

Species	Moisture (per cent.)	Glue Adhesion			
		Dry		Hot wet	
		Failing load (lb.)	Glue failure (per cent.)	Failing load (lb.)	Glue failure (per cent.)
Dichopsis elliptica (pali)	6.44	263	6	300	12
Mangifera indica (mango)	6.05	287	0	284	32
Dysoxylum malabaricum (white cedar)	9.81	295	0	316	20
Canarium strictum (dhup)	5.85	219	20	319	54
Zanthoxylum rhetsa (mullilam)	5.36	259	63	301	97
Canarium (dhup)	—	297	0	228	0
Dysoxylum malabaricum (white cedar)	—	290	0	230	0
Canarium sp. (dhup)	—	210	95	132	55
Dysoxylum malabaricum (white cedar)	—	178	100	135	75
Canarium sp. (dhup)	—	307	0	289	23

TABLE 9. (*Continued*)

Dysoxylum malabaricum (white cedar)	—	241	50	187	100
Canarium sp. (dhup)	—	347	45	200	100
Dysoxylum malabaricum (white cedar)	—	170	40	—	—
Dichopsis elliptica (pali)	10.00	473	70	443	80
Mangifera indica (mango)‡	10.50	339	50	239	90
Dysoxylum malabaricum (white cedar)	8.50	369	80	321	50
Zanthoxylum rhetsa (mullilam)‡	8.50	388	80	285	100
B. S. 1203*	—	250	—	100	—

* B.S. 1203 calls for dry strength of 250 lb./sq. in. and a hot wet strength of 100 lb./sq. in. after 3 hours' boiling

‡ Data obtained with phenol—formaldehyde resin used as wood adhesive by D. Narayanamurti and Kartar Singh (1944)

Adhesives and Moulding Powders from Coconut Shells

Finely powdered coconut shells when subjected to a thermal hydrolysis process at about 195°C., as described earlier and then treated with 15 per cent. of its weight of phenol at 140° to 150°C gave a reaction product which when made into a slurry in alcohol gave a satisfactory plywood thermosetting adhesive. The curing conditions were found to be 165°-175°C. and 250-300 lb./sq. in. for 15 minutes. Table 9 gives the results of tests on plywood prepared with the adhesive.

It was also noticed that the thermally activated material could be moulded at about 2000 lb./sq. in. at 150-175°C. It was thermoplastic and was found to tolerate fillers. Moulded products could be obtained with sawdust as a filler (Table 10).*

TABLE 10. MOULDED PRODUCTS FROM ACTIVATED COCONUT SHELLS WITH SAWDUST AS A FILLER

Material	Sp. gr.	Moisture (per cent.)	Modulus of rupture (lb./sq. in.)	Water absorption (per cent.)	Swelling (per cent.)
Activated coconut shell	1.37	4.38	1135	2.64	0.0
-do-chir sawdust (1:1)	1.42	6.12	4735	3.72	2.54
-do- (3:2)	1.37	5.62	3682	3.56	2.18
-do-bamboo dust (1:1)	1.30	5.39	5625	3.50	1.54
-do- white cedar sawdust (1:1)	1.44	5.62	6385	3.52	1.33
C.N. shells with phenol and coconut coir as filler	1.38	6.37	8012	6.37	3.89

Furfural from Agricultural Wastes

As can be seen from the results of analysis reported in Table 1, most of the wastes are rich in pentosan and thus can be considered

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*Process patented.

TABLE 11. LIGNOCELLULOSIC FLOURS AS FILLERS IN PHENOLIC PLASTICS

Physical properties of boards — Temperature of pressing : 160°C — Time of pressing : 30 minutes

Filler	Resin (per cent.)	Pressing conditions		Moisture of board (per cent.)	Sp. gr.	Flexural strength (lb /sq. in.)	Moisture absorption (per cent.)		Swelling in thickness (per cent.)	
		Temp. (°C.)	Pressure (lb./sq. in.)				24 hrs.	7 days	24 hrs.	7 days
Spent tea leaves	5	160	2000	8.2	1.365	2430	16.1	32.0	13.6	29.5
	15	,,	,,	5.5	1.390	5653	3.0	8.5	1.78	3.5
	25	,,	,,	5.0	1.400	5808	2.4	6.5	0.6	2.1
	40	,,	,,	4.5	1.400	5340	1.8	3.5	0.43	2.0
Spent tea leaves 20 per cent. white cedar sawdust	10	150	,,	7.2	1.424	4540	5.1	19.3	9.0	23.3
Spent tea leaves 1/32 in. veneer faced	10	,,	,,	8.2	1.450	18720	5.7	14.8	6.3	13.5
Powdered coconut shells 80 mesh	10	160	,,	2.1	1.424	6643	0.3	1.0	5.4	5.4
Powdered arecanut husk	10	160	200	6.3	1.367	4980	3.3	8.5	6.3	11.7

TABLE 12. ACID HYDROLYSED MATERIALS AS FILLERS IN PHENOLIC PLASTICS

Filler	Pressing pressure (lb./sq. in.)	Moisture of board (per cent.)	Sp. gr.	Flexural strength (lb./sq. in.)	Moisture absorption (per cent.)		Swelling in thickness (per cent.)	
					24 hrs.	7 days	24 hrs.	7 days
Arecanut husk	2000	6.3	1.35	8300	3.8	5.5	6.5	6.5
	,,	3.5	1.38	8500	0.25	0.3	3.0	3.0
	,,	3.5	1.39	8600	0.3	0.5	2.7	3.0
Coconut shells	3000	5.5	1.41	8300	0.7	1.0	5.0	5.0
	,,	5.0	1.39	7850	0.4	0.4	3.0	3.0

as potential sources of furfural. Corn cobs, oat hulls, etc., are used for this purpose in the U. S. A. Some trials were done with coconut shells and arecanut husk both in the laboratory and on a pilot plant scale. The influence of various factors like the pressure and temperature of digestion, catalyst concentration, period of digestion, liquid/solid ratio were investigated in laboratory studies. Further work is necessary for coming to definite conclusions. The original raw materials as well as the ligno-cellulosic residues derived from them in the laboratory and pilot plant studies of furfural production were tried as filler in a phenolic resin. The results of tests on the moulded products are given in Table 11.

From the Table 12, it can be seen that many agricultural wastes could be turned into useful products. But in order to develop the economic utilization of these residues as pointed out by Clark and Lathrup, it is necessary to establish sound economic methods for the collection or purchase and preservation of these materials combined with a policy of sound merchandising and business management.

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