Industrial Charcoal and Chemicals From Ipil Ipil Wood

by

E. P. VILLANUEVA
& N. B. LAXAMANA[1]

SUMMARY

Pilot-plant studies on the carbonization of ipil-ipil wood indicated the possibility of producing an industrial-grade charcoal containing 87.23 per cent fixed carbon, 10.45 per cent volatiles, and 2.32 per cent ash. The charcoal has a heating value of 7470 calories per gram.

Ipil-ipil wood yielded about 9.21 per cent wood tar and wood-tar oils, 25.46 per cent pyroligneous liquor and 34.42 per cent charcoal. The pyroligneous liquor recovered contained 1.76 per cent acetic acid, 0.81 per cent methanol and 3.73 per cent soluble tar, based on oven-dry wood.

INTRODUCTION

There is perhaps no other Philippine tree as common as ipil-ipil (*Leucaena leucocephala* (Lam.) de Wit). It could be found in practically every part of the country (10)[2]. It grows sturdily and abundantly in any condition, whether it be in the fertile soils of the farms and valleys or in the rocky cliffs of hills and riverbanks. Ipil-ipil could well be the Philippine wonder wood for charcoal production because despite its comparatively fast growth and short rotation age, the charcoal produced from its wood is hard and heavy.

A hard, heavy charcoal which meets the limiting requirements for volatiles is applicable for industrial purposes. According to Beglinger (2), unlike most other industrial raw materials, charcoal is seldom sold on specifications. The usual market guarantees relate only to the weight per bushel and to the percentage of contained volatile matter. Limiting values for volatile matter and for moisture are that they be not more than 14 per cent and 2 per cent, respectively (2).

The Forests Commission of Victoria (5) theorized that the ultimate test of charcoal quality is its performance in a gas producer. W. Atkinson Wood (5) showed that a suitable producer-gas charcoal when burnt, should not cause blackening of a tin plate placed directly over the embers. Laboratory tests at the Institute showed that this corresponds to charcoal containing volatiles less than 20 per cent.

The volatiles are chemicals which are formed during the carbonization process. They escape from the burning wood as smoke. When ipil-ipil wood is carbonized, about 60 per cent of its original weight is lost as smoke (1). However, when the vapors are passed through a condenser, a liquid product called *pyroligneous liquor* and a black, waxy residue called *settled tar* are recovered.

The chief components of the pyroligneous liquor are acetic acid, methanol and soluble tar (11). The commercial recovery of these chemicals have largely augmented the wood

[1] Forest Products Technologist and Jr. Forest Products Technologist, respectively, Forest Products Research Institute, College, Laguna.

[2] Numbers in parentheses refer to Literature Cited.

distillation industry of the United States and Germany during World War II (7, 11). Acetic acid is presently, one of the chief chemical imports of the Philippines (3).

The settled tar contains wood-tar oils (4). It is not definitely known how wood tar and wood-tar oils are used in Philippine industry but they are imported into the country in large quantities. In 1962, 1,047 tons worth ₱238,000.00 were imported. Until lately, there is an apparent increasing price trend of this product. In June 1963, 163 tons worth ₱53,-000.00 were imported, and in July 1963, only 33 tons came into the country but at a total cost of ₱87,000.00 (3).

The operation of some Philippine industries depends on wood-carbonization products. Certain metallurgical, chemical and home industries need industrial-grade charcoal as fuel and raw material (Table 1). The manufactures of plastics, textiles, paints, rubber and pharmaceutical products need acetic acid, methanol and probably wood tar and wood-tar oils.

This study is therefore aimed to develop a process of producing an industrial-grade charcoal from ipil-ipil wood. Experiments were conducted to evaluate the smoke chemicals in terms of its acetic acid, methanol, wood tar and wood-tar oil contents.

EXPERIMENTAL

Raw Material

Matured ipil-ipil trees obtained from the Mt. Makiling area, Los Baños, Laguna were used in the study.

Round woods were split and cut to uniform sizes, about 2 in. x 2 in. x 12 in. and air-dried. The average moisture content of the wood charge in every run was determined.

The wood samples, analyzed according to the methods suggested by the Technical Association of the Pulp and Paper Industry (12), showed the following chemical compositions:[3]

(a) Ash	2.25%
(b) Alcohol-benzene extract	5.09%
(c) Hot-water extract (w/o prior leaching)	11.60%
(d) Hot-water extract (leached)	6.95%
(e) Lignin (corrected for ash)	23.90%
(f) Solubility in 1% NaOH	16.35%
(g) Pentosan	15.24%
(h) Holocellulose (by difference)	62.17%

Carbonization

The carbonization runs were made in a stainless steel, electrically-heated retort, equipped with a water-cooled condenser (Fig. 1). At tight-fitting steel cover was used to seal the wood charge completely in the coaling chamber.

The retort was fitted with a temperature-recorder controller. In all runs, this was set at 900 deg. F to allow carbonization to proceed as fast as possible. Three thermocouples extending into the coaling chamber were fitted at strategic positions, so that the temperature in every part of the chamber may be determined (Fig. 2).

Heating was terminated when no more smoke came out of the flue. At the completion of a run, cooling water was allowed to continue flowing through the condenser for another half hour before the control valves for hot gases and water were closed.

The weight of the condensate was determined 15 hours after heating was stopped. The settled tar was separated from the pyroligneous liquor by settling and decantation. After cooling for 20 hours, the steel cover was opened and charcoal yield was determined.

[3] Percentages are on oven-dry weight basis.

Pyroligneous Liquor Analysis

1. *Methanol.* — The methanol content was determined by the specific gravity method.

2. *Total acids.* — The total acid content of the pyroligneous liquor was determined on a redistilled sample as acetic acid by direct titration with standard base using phenolpthalein as indicator.

3. *Soluble tar.* — The black residue left in the flask after redistilling the clear pyroligneous liquor was considered as soluble tar. The weight of soluble tar was obtained by difference.

Chemical Analysis and Calorimetric Determinations

Chemical analyses of the charcoals were made according to the methods of Moore and Beglinger (8). The amount of volatile matter was reported as the percentage of loss in weight of oven-dry charcoal after heating for 11 minutes in a covered crucible at 950 deg. C. The weight of residue, after completely burning the charcoal for 6 hours at 750 deg. C, was reported as the ash content of the charcoal. The fixed carbon content was obtained by difference.

A Parr peroxide bomb calorimeter was used in determining the heating value of the charcoals (6, 9).

Redistillation of Settled Tar

The settled tars were subjected to a secondary distillation in Pyrex distilling flasks at atmospheric pressure. By careful, controlled heating, the distillation-temperature schedules below were followed:

DISTILLATION TEMPERATURE RANGE, DEGREES C	APPROXIMATE DURATION, HOURS
Room temperature to 210	1.5
210 to 235	1.0
235 to 270	1.0
270 to 315	1.5

The distillates at different temperature ranges were collected separately. The color and other physical properties of the liquids were observed. Their specific gravities, using the pycnometer method, were determined.

RESULTS AND DISCUSSIONS

Charcoal Yield and Quality

The carbonization process used in this study was based on a controlled pyrolysis at a temperature range of 800 to 900 deg. F. The heat generated at this range seemed sufficient to carbonize the wood and "refine" the charcoal by vaporizing the complex organic chemicals, thereby leaving a product rich in carbon content. The maximum temperature was low enough to eliminate the danger of incurring unnecessary deterioration on the equipment.

A charcoal yield of 34.42 per cent was obtained at the above conditions (Table 2). The charcoal analysis of 87.23 per cent fixed carbon and 10.45 per cent volatiles (Table 3) substantially met the limiting chemical requirements of industrial-grade charcoal. Its heating value of 7470 calories per gram compared favorably with coke and the industrial "white charcoal" of Japan (Table 6).

The indicated carbonization process has produced a hard and dense but cracked charcoal. The ruptures probably resulted from rapid drying coupled with the violent expulsion of hot gases from the interior of the wood during carbonization. However, this condition has not affected the utility of the products to an appreciable extent.

Investigations on the Pyroligneous Liquor

The composite sample of pyroligneous liquor, obtained from the carbonization of ipil-ipil wood, was a turbid, brown liquid having a distinctly pungent odor. It contained a dye which left a dirty brown stain on the hands and clothes. The stain cannot be removed by rinsing with ordinary soap and water.

On an oven-dry wood basis, about 25.46 per cent pyroligneous liquor was obtained (Table 2). The chemical analysis of this liquid indicated that the carbonization of one ton of oven-dry ipil-ipil wood would yield approximately 17.60 kilos acetic acid, 8.10 kilos methanol, and 37.30 kilos soluble tar (Table 4).

Wood Tar and Wood-Tar Oils

Laboratory studies at the Institute showed that one ton of oven-dry wood could produce 90.20 kilos of wood tar. This tar contained 7.14 per cent light oils (specific gravity range 0.90 to 1.0) distilling below 200 deg. C and 9.91 per cent heavy oils (specific gravity range 1.01 to 1.03) distilling from 200 deg. C to 315 deg. C (Table 5). The greater fraction of wood tar is pitch.

CONCLUSION

1. The carbonization of ipil-ipil wood, at the conditions used in this study, produced charcoal that may be suitable for industrial application. This charcoal had an average volatile matter content of 10.45 per cent, which is much lower than the limiting value of the contained-volatile-matter requirement of industrial-grade charcoal.

2. If the smoke that escapes during the carbonization of ipil-ipil wood is condensed, it is possible to recover 254 kilos pyroligneous liquor and 92 kilos wood tar and wood-tar oils, per ton of oven-dry wood.

3. The pyroligneous liquor may be processed to yield about 17.60 kilos acetic acid and 8.10 kilos methanol, per ton of oven-dry wood.

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Table 1. *Some common industrial outlets for charcoal in the form of lumps, screenings, powder and briquettes.*

Metallurgical

Aluminum metal
Armor plate
Case hardening
Cobalt metal
Copper, brass and bronze
Electro manganese
Foundry molds
Magnesium metal
Mining
Molybdenum
Nickel
Pig Iron
Powdered iron
Special alloys
Steel

Specialized Fuel

Citrus growers
Foundries
Meat and fish curing
Tobacco curing

Chemical

Activated carbon
Black powder
Brake linings
Carbon disulfide
Carbon monoxide
Catalyst reactor
Electrodes
Fertilizer
Galvanizing
Gas cylinders
Glass
Glues
Graphite
Molding resins
Nursery mulch
Pharmaceuticals
Plastics
Poultry and stock feeds
Potassium cyanide
Rubber
Sodium cyanide

Source: Beglinger, E. 1952. Charcoal production. U. S. Forest Products Laboratory Report No. R1666-11. Madison, Wisconsin.

Table 2. *Carbonization products of ipil-ipil wood*

NOTE: All samples were carbonized in an electrically-heated retort at a maximum temperature of 900 deg. F. The carbonization time was 8 hours.

Sample	Moisture content[a] per cent	Oven-dry Weight of wood kg.	Charcoal[b] per cent	Pyroligneous liquor[c] per cent	Settled tar[b] per cent	Uncondensible gases and losses[d] per cent
A	22.87	17.49	36.30	20.89	4.17	38.64
B	17.80	15.04	31.00	22.40	6.05	40.55
C	25.30	13.56	33.82	30.60	6.25	29.33
D	25.00	16.50	36.81	28.60	5.35	29.24
E	23.51	15.36	34.20	24.80	5.60	35.40
Average	22.90	15.59	34.42	25.46	5.48	34.64

[a] Percentage was based on the weight of wood as received.
[b] Percentage was based on the oven-dry weight of wood charge.
[c] The weight of water in the wood charge was subtracted from the total weight of liquid product recovered.
[d] Percentage of uncondensible gases and losses = [100 — (% charcoal ÷ % pyroligneous liquor ÷ % settled tar).]

Table 3. *Proximate chemical analysis of charcoal of ipil-ipil wood*[e]

Sample	Moisture[f] per cent	Volatile matter[g] per cent	Ash[g] per cent	Fixed carbon[g] per cent
A	0.44	9.20	1.93	88.87
B	5.00[h]	13.20	2.30	84.60
C	1.74	10.58	2.78	86.64
D	4.78[h]	8.80	2.46	88.74
E	0.97	10.45	2.12	87.43
Average	—	10.45	2.32	87.23

[e] Unless otherwise stated, the charcoal samples were obtained immediately after opening the retort.
[f] Percentage was based on charcoal as received.
[g] Percentage was based on moisture-free charcoal.
[h] Charcoal samples were obtained after a stabilization period of at least 24 hours at room temperature.

Table 4. *Pyroligneous liquor analysis*[i]

Samples	Methanol per cent	Acidity as acetic acid per cent	Soluble tar per cent
A	0.65	1.28	3.66
B	0.74	1.32	3.50
C	0.86	2.16	3.56
D	0.96	2.30	3.94
E	0.84	1.74	4.01
Average	0.81	1.76	3.73

[i] All percentages are based on the oven-dry weight of wood charge.

Table 5. *Fractional distillation of settled tar*[j]

Temperature range deg. C	Average weight Percentage of fraction	Average specific Gravity of fraction	REMARKS
150—210	3.41	0.913	straw-yellow oil
210—235	3.73	1.010	orange oil
235—270	4.76	1.025	reddish-orange oil
270—315	5.15	1.031	reddish-brown oil
(Residue)	64.40	xxxxx	waxy, black oil

[j] All percentages are based on the weight of settled tar.

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tractives, and other important characteristics of potential hybrids should be known through an effective research of the institute. The wood-using industries should also conduct studies on the quality and saleability of the finished products, together with the economics of the improved varieties and species, like the inbreds and hybrids. An alternative but a philanthropic contribution would be a financial support to the various projects within the scope of the program.

SUMMARY

The Philippines today is destroying its forest faster than any country in the world. Illegal kaiñgin, timber smuggling, and indiscriminate logging have drained much of the forest resource. However, no immediate measures of tree improvement are taken to check this critical forest problem. Agriculture has long developed high-yielding varieties of rice, corn, abaca, and other crops but practically no improved tree varieties or hybrids could be brought to light yet, after more than half a century of forestry practice in the Philippines.

A solution to the foreseeable problem of timber shortage has been proposed through the implementation of a well-planned forest tree improvement research. This involves studies on the genetic and silvicultural aspects of improving the quality of existing tree species that are of commercial importance. Efforts would be exerted to create a population of genetically superior trees that are expected to answer the dire needs of the wood-using industries. Attainment of this objective calls for the production of hybrids that are characteristically fast-growing, resistant to pests and diseases, of wider ecological adaptation, and superior wood quality. The castle of this dream could be built up through the cooperative efforts of the various forestry agencies that would undertake the different phases of the research program. Most possibly, the creation of a Committee on Forest Tree Improvement could be an incendiary step to keep the ball rolling. To begin with, the committee would explore the possibilities of securing the necessary research fund which is considered as the lifeblood of the proposed undertakings.

On the whole, the time is already ripe; hence, the Filipino Foresters should appropriately act now in order to create and perpetuate better trees for tomorrow and a greener Philippine forest.

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Table 6. *Calorific value of coke, coking coal and wood charcoals*

Kind of Fuel	Volatile matter per cent[k]	Fixed carbon per cent[k]	Heating value per cent[k]
Coking coal[l]	19.0	76.0	7500
Coke[l]	5.0	87.0	7100
Black charcoal (Japan)[m]	12.0	86.0	7158
White charcoal (Japan)[m]	5.0	93.0	7235
Spruce wood charcoal (U.S.A.)[l]	14.4	83.6	7310
Coconut shell charcoal (Philippines)[n]	18.8	77.4	6700
Ipil-ipil wood charcoal (Philippines)[o]	11.0	86.7	7472

[k] Based on moisture-free charcoal.
[l] Anonymous. 1955. Wood burning. Food and Agricultural Organization of the United Nations, Rome, Italy. p. 4
[m] Kishimoto, S. 1961. Firewood and charcoal. Chemical utilization of wood. Overseas Technical Cooperation Agency, Ministry of Agriculture and Forestry, Japan. pp. 272-273.
[n] Coconut shell charcoal was obtained from a commercial sample.
[o] Average of 6 distillation runs. The calorific values of coconut shell and ipil-ipil charcoals were determined by the sodium peroxide method at the Institute.