

Double Pyrolysis of Chrome Tanned Leather Solid Waste for Safe Disposal and Products Recovery

Mr. C. Sethuraman¹, Mr. Kota Srinivas², Dr. G. Sekaran³

Abstract— Disposal of chrome tanned leather waste (CTLW) generated in leather processing industries has many environmental implications. This paper presents a double pyrolysis (PI & PII) method to dispose hazardous chrome tanned leather solid waste by converting it into useful products. When CTLW was subjected to double pyrolysis, three major products obtained were; (i) an energy enriched combustible gas (32.01% - PI & 42.45% - PII); (ii) a high fraction condensate (33.32% (PI & PII)) and (iii) a carbonaceous residual ash containing trivalent chromium (34.67%- PI & 24.23% -PII). In addition, 2.73% of liquid fuel, 75.24% of nitrogen enriched liquid and 22.03% distillate tar as sub-products from the high fraction condensate. SEM, GC-MS, ¹H & ¹³C NMR and TGA revealed that the products were free from toxic chromium species and contains both aliphatic and aromatic hydrocarbons. The residual ash displayed high thermal stability when run under nitrogen atmosphere (simulating 1st pyrolysis). The observations on generation of no toxic compounds upon double pyrolysis, suggests the suitability of this process towards achieving safe disposal of hazardous leather solid waste and to recover useful products.

Index Terms— Chromium, Double pyrolysis, Energy recovery, Fuel gas, Leather Waste, Tannery, Zero-discharge

1. INTRODUCTION

Leather industry processes 6.8 million tons of wet salted hides and skins worldwide in a year. It generates about 75-80% of solid wastes during the process, in which, variety of chemicals used to convert putrescible collagen fibres into non putrescible leather finished products. One ton of wet salted hides would yield only 195 kg of grain and 60 kg of split: a total yield of 255kg of finished leather and the remaining in the form of wastes such as fleshing, trimmings, unusable chrome split, wet blue trimmings, crust leather waste, chrome shaving, buffing dust and finished leather off-cuts [1].

Basic chromium sulphate (BCS) is the most widely used tanning material for converting putrescible collagen fibres into non putrescible leather matrix. Only 60% of chromium salts applied for tanning process react with the raw materials and the rest of chromium salts disposed along with the waste material and also into the waste water [2]. Amongst the waste, CTLW is a proteinous fine particulate solid waste impregnated with chromium, synthetic fat, oil, tanning agents and dye chemicals. About 2-6 kg of CTLW is liberated as a solid waste per ton of skin/hide processed [3].

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The energy content of tannery leather solid waste is more than 50% in comparison to hard coal, is normally 20 MJ/kg in dry basis [4]. The chromium content in solid leather waste (wet blue leather), is approximately 30g kg⁻¹ (w/w) [5]. Brazilian Environmental Council (CONAMA) classified chromium containing leather waste material as a category-one waste, one of the most dangerous and harmful wastes if discarded into the environment without any further treatment [6].

Chromium containing leather waste is carcinogenic in nature and it causes clinical problems like respiratory tract ailments, ulcers, perforated nasal septum, kidney malfunction [7] and lung cancer [8]. Because of this, such a material needs a special disposal, which is very expensive [9]. In the absence of any economically viable technology to dispose the solid leather waste, land co-disposal, thermal incineration and anaerobic digestion methods are currently being practiced [10]. Current practising methods of disposing these wastes have the following disadvantages:

The available landfill sites rapidly reach their total capacity and the authorization of new sites becomes difficult [11]. The improper and the manual handling and transfer of leather waste in open vehicles create unhygienic conditions. Disposal of waste in low-lying areas without proper liners allow the leachate to mix with ground water causing water contamination.

It has been reported [12] that during thermal incineration at 800°C, Cr³⁺ was converted to Cr⁶⁺ by 40%. Thermal incineration causes serious air pollution problems due to emission of toxic hexavalent chromium (Cr⁶⁺), halogenated organic compounds, poly aromatic hydrocarbons etc. into environment. The major species formed from Cr³⁺ during thermal incineration of solid waste are Cr₂(SO₄)_{3(s)}, CrOCl_{2(g)} and Cr₂O_{3(s)} which later transformed into Cr⁶⁺ [13]. Hexavalent chromium (Cr⁶⁺) is mobile in

the environment and is highly toxic. It can penetrate the cell wall and exert its noxious influence in the cell itself, being also a source of various cancer diseases [14]. At short-term exposure levels above the maximum contamination level, Cr⁶⁺ causes skin and stomach irritation or ulceration. Long-term exposure at levels above the maximum contamination can cause dermatitis, damage to liver, kidney circulation, nerve tissue damage and death [15, 16]. Air pollution is created by odour nuisances and the generation of green house gases from most of the landfill sites. Investments cost on anaerobic digestion plant is very high and also it does not provide a solution for zero waste disposal.

An earlier study on leather wastes suggested that pyrolysis experiments on leather wastes at 450 °C and 600 °C using fixed bed reactor loaded with 50-60 g, yielded the charred residue and ammonium carbonate besides gas and oil products [17]. It was stated that the carbonaceous residue (chars) resulted was in the range 37.5%-48.5% and their calorific value was in the range 4300-6000 kcal/kg, suitable for use as solid fuel. The heat content of the gas was not tested and concluded that waste leather is a useful recycling resource and the conversion of wastes into activated carbon and fuels may be recognized as an attractive approach and further works are necessary before large-scale application.

The encouraging statement of Yilmaz et al (2007) was the lead for the present study and based on the above summarized drawbacks and the necessity on management of chromium containing leather solid wastes. The present study focussed the management of chromium containing leather wastes through double pyrolysis. Like incineration, pyrolysis is also a thermal process that uses high temperatures to break down the organic waste without the participation of oxygen. The advantage of pyrolysis over combustion is reduction in CO₂ which play a vital role in green house effect. In single pyrolysis system, percentage of residual carbonaceous material (40%) needs additional treatment system, which is a major drawback observed from our earlier experiments.

Hence, a double pyrolysis approach was used in this study to assess the mass of the residual carbonaceous material, gases and value added products. The by-products obtained from the process were further subjected to chemical and analytical methods.

2. MATERIALS AND METHODS

2.1 Characterization of Chrome Tanned Leather Waste

CTLW was characterised for moisture content, volatile matter, carbon, ash, chromium, fat, protein content, calorific value etc., using standard procedures and the results are given in table-1.

TABLE 1
CHARACTERIZATION OF CTLW

Parameters	Value
Proximate Analysis (wt %)	
Moisture %	5.50
Fixed Carbon#	26.62
Volatile Matter	60.30
Ash	7.58
Ultimate Analysis (wt %)	
Carbon	55.31
Hydrogen	7.86
Nitrogen	12.56
Sulphur	4.68
Oxygen	8.54
Other Analysis	
Fat (dry wt %)	7.46
Protein content	80.63
pH of soluble matter	4.67
Chromium	2.8
Density (g/cm ³)	0.260
GCV (Kcal/kg)	4246

By Difference

2.2 Double pyrolysis process and system description

The main parts of the double pyrolysis system are i) furnace for raw feed and residual carbon for second pyrolysis ii) reactor stills iii) microprocessor based temperature indicator controller iv) digital energy meter v) condenser vi) gas filter vii) syngas blower viii) water circulation pump ix) scrubber x) gas flow chamber vi) distillation unit as shown in the following process flow diagram (Fig. 1). The outer chamber of the furnace was fabricated using mild steel with necessary reinforcement for mechanical rigidity. The outer dimensions of the furnace were length, 650mm; breadth, 650mm and height, 450mm. The reactor still of first pyrolysis was made up of SS316 grade with inner diameter, 150mm; height, 300mm; with wall thickness, 5mm. The secondary pyrolysis reactor still was also made up of SS316 grade with inner diameter, 80mm; height, 200mm; with wall thickness, 5mm.

The waste material loaded in the reactor still heated in the box type furnace using A1 grade kanthal heating coils, temperature sensor, chrome/alumel ("K" type) with single phase 230V A.C. operated by micro processor based temperature indicator controller using thyristors. This electrically operated furnace works based on resistive heating principle. Due to the resistive property of the Kanthal coils, power fed to the elements are converted to heat energy. This heats up the inner chamber of the furnace where the CTLW samples are placed as per the temperature programming set up to 800 °C. The volatile matter generated from the reactor enters into trimetallic nano-catalytic converter (NCC) to enhance the combustion properties of the gas.

The liquid oil collected from NCC goes to distillation unit where the by-products viz. i) thiol containing diesel like fuel, ii) ammonium containing fertilizer liquid, iii) ammonium carbonate salt is produced. The gas which is free from liquid oil passes through the gas filter, scrubber, gas flow chamber and got cleaned. The cleaned gas from gas flow chamber can be

used for both thermal and electrical applications. The volatile matter removed residual taken out from the first pyrolysis reactor still then transformed into secondary pyrolysis reactor still and heated upto 900°C to remove the left over volatile matter present in the residue after first pyrolysis.

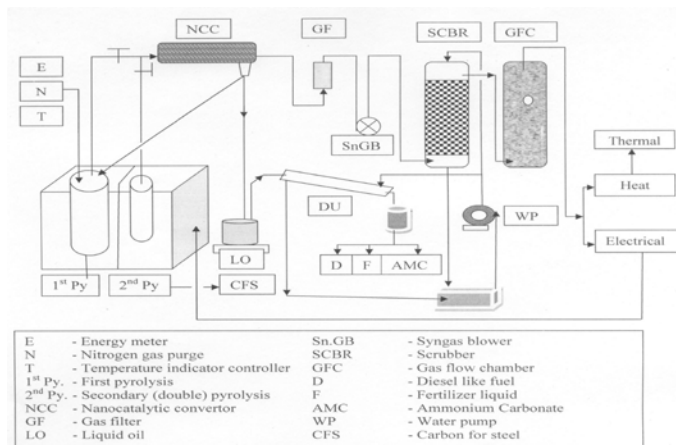


Fig. 1. Process flow

2.3 Fabrication of Pyrolysis Furnace

Considering the input quantity of 2 kg load and material physiochemical properties, the batch type double pyrolysis gasification system was designed as per the specification given below:

Input quantity in mass, m = 2 kg
 Specific heat of chrome tanned leather, 10% moisture, Cp = 0.35 cal/g/°C
 Density of CTLW = 0.38 g/cm³
 Volume of reactor = 2000 / 0.38 = 5263 cm³
 Internal diameter of reactor = 15cm
 Length of the reactor = 5263 / (3.142 x 7.5 x 7.5) = 29.77 cm
 Thickness of reactor vessel, t = 3mm
 Weight of the reactor vessel = 10.30kg
 The heat to rise the 10.30g SS 316 grade material from ambient (30°C to 800°C), its specific heat is 0.38 cal/g/°C = 10.30 x 0.38 x (800-30) = 3013.78kcal = 3013.78 / 860 = 3.5 kW (3500W)
 Interm of power, P = 3.5 kW (3500W)
 This power can be met with single phase (i.e. Voltage at 230V), the required current to be passed through the resistive heating coil is = P / V = 3500 / 230 = 15.21A
 Resistance of the coil, R = 230 / 15.21 = 15.12 Ω
 Electrical heating coils, 12 numbers, each 300W; 2Ω resistance, 12 coils kept inside the ceramic tubes, connected in series (8 coils in service and 4 coils kept as spare), temperature sensor insulated with glasswool is shown in Fig. 2 (a, b). The heating capacity of 8 coils is 2400W. Depents upon the requirement of rate of heating all 12 coils could be connected in series to

achieve 3600W.

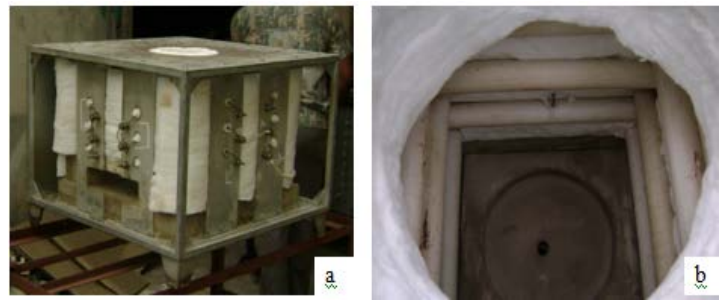


Fig. 2 Fabrication of pyrolysis furnace a) connected with 12 coils, b) temperature sensor with glasswool insulated ceramic tubes.

2.4 Instrumental Analysis

The elemental compositions interms of Carbon, Hydrogen, Nitrogen and Sulphur (CHNS) of raw CTLW, residual carbon obtained from first and second pyrolysis, liquid oil were determined using Elementar-Vario Micro Cube analyser. Thermo-Gravimetric Analysis (TGA) was carried out to assess the weight loss as a function of temperature under nitrogen atmosphere using Universal TGA Q50 V20.6 Build 31. The loss of mass of the residual ash also carried out under air atmosphere using TGA Q500 V20.10 Build 36 to study the effect of air in mass reduction.

Scanning Electron Microscopy (SEM) analysis was carried out using Hitachi S-3400N to determine the surface morphology of the samples with high resolution. Energy Dispersive X-ray analysis (EDAX) was carried out on raw CTLW, residual ash and liquid oil to determine the elemental composition. Fourier Transform Infrared (FTIR) spectroscopy analysis was carried out using Thermo Nicolet, model 330 to determine the functional groups present in the samples. The hexavalent chromium (Cr⁶⁺) content in the residual carbon of first and secondary pyrolysis, and in liquid oil were determined calorimetrically by alkali digestion using NaOH, Na₂CO₃, MnSO₄ in the presence of phosphate buffer (K₂HPO₄ and KH₂PO₄). The Cr total content was determined by acid digestion using strong oxidising agent and followed by reaction with diphenyl carbazide.

3 RESULTS AND DISCUSSION

3.1 Characteristics of CTSW

The CTLW was characterised for moisture content, 5.50%; protein content, 80.63%; and pH, 4.67. The composition of CTLW as per ultimate analysis was Carbon, 55.31%; Hydrogen, 7.86%; Nitrogen, 12.56%; Sulphur, 4.68%; Oxygen, 8.54%. The proximate analysis showed the volatile matter, 60.30%; ash, 7.58% and fixed carbon, 26.62%. The density of CTLW was 260kg/m³ and Gross Calorific Value was 4246kcal/kg.

3.2 Thermo gravimetric analysis (TGA)

The CTLW sample (weight 2.83mg) was heated in a platinum pan from 30 to 800°C at the rate of 20°C/min. The TGA spectrum of CTLW under nitrogen atmosphere (Fig. 3a) shows that the weight loss was only 5% upto 222.23°C. There was a steady decrease in weight of the sample by 56.22% upto

481.52°C and this may be attributed to the elimination of surface bound water molecules and volatile organic compounds from the matrix. There was a further reduction in mass by 8.61% on heating from 481.52°C to 789.83°C due to the thermal decomposition/ volatilization of the peptide compounds in which carbon constitutes the back bone of the chain and attached with functional groups viz hydrogen, nitrogen containing group, carboxylic acid group. The side attached to the main carbon chain volatilized at higher temperature which resulted in the reduction in mass by about 65% at 800°C. The TGA of the CTLW suggests that the volatile organic compounds were destructed upto 500°C using the combined oxygen of 26.77% in it. The residual ash resulted during destructive distillation of CTLW under nitrogen atmosphere (simulating 1st pyrolysis process) was 35.17%. The TGA of residual ash under nitrogen atmosphere (simulating 1st pyrolysis process) shows that the material was thermally stable upto 791.80°C since the mass of unburned residual ash was 81.69% (Fig. 3b), this suggests that very little change in mass of residual ash alone would be resulted under 1st pyrolysis condition. The TGA of residual ash obtained under double pyrolysis (Fig. 3c) shows that the mass of unburned residual ash was 45.96% at 800°C. It indicates that double pyrolysis could reduce the mass of the unburned residual ash from 81.69% to 45.96% which may be due to the delinking of peptide bonds and formation of carbon attached gaseous compounds at high temperature. The TGA of condensate oil (Fig. 3d) shows that the solid residue left at 296.45°C was 11.68% and it was reduced to 3.586% at 791.06°C, indicating that the condensate oil has more than 96% of volatile components.

Fig. 3. TGA spectra of a. CTLW under nitrogen atmosphere, b. residual ash under 1st pyrolysis, c. residual ash under double pyrolysis and d. condensate oil

3.3 Analysis of SEM

The SEM image of CTLW (Fig. 4, a) shows the isolated collagen fibres. This is due to the presence of fat liquors that surrounded the fibres causing their separation. SEM analysis of residual ash presented in figure (Fig. 4, b) shows that 1st pyrolysed state of collagen fibres as separated carbon slabs. SEM image of double pyrolysis residual ash (Fig. 4, c) indicates that the carbon slabs were reduced to the flakes of ash with inorganic chromium spreading as white granules over it. However, the SEM analysis of distillate as shown in figure (Fig. 4,d) indicates that the condensate oil is not pure homogeneous, it is admixed with other substances.

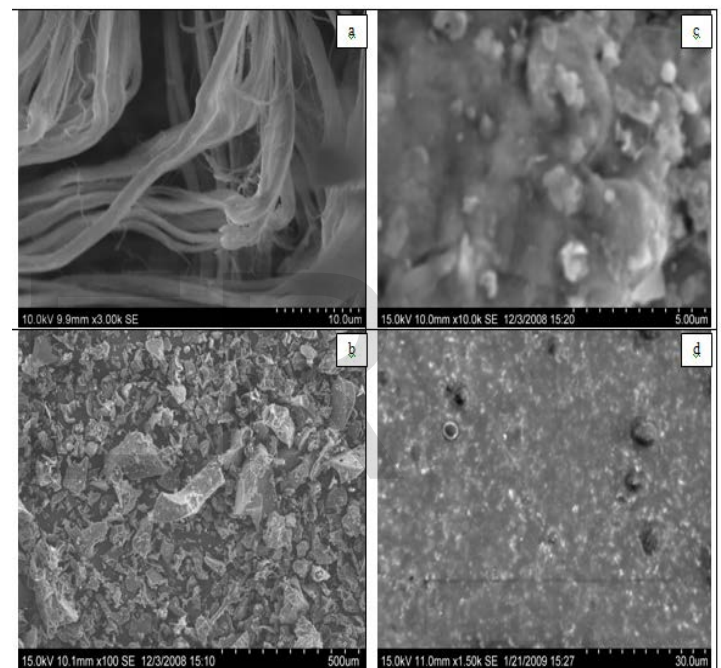
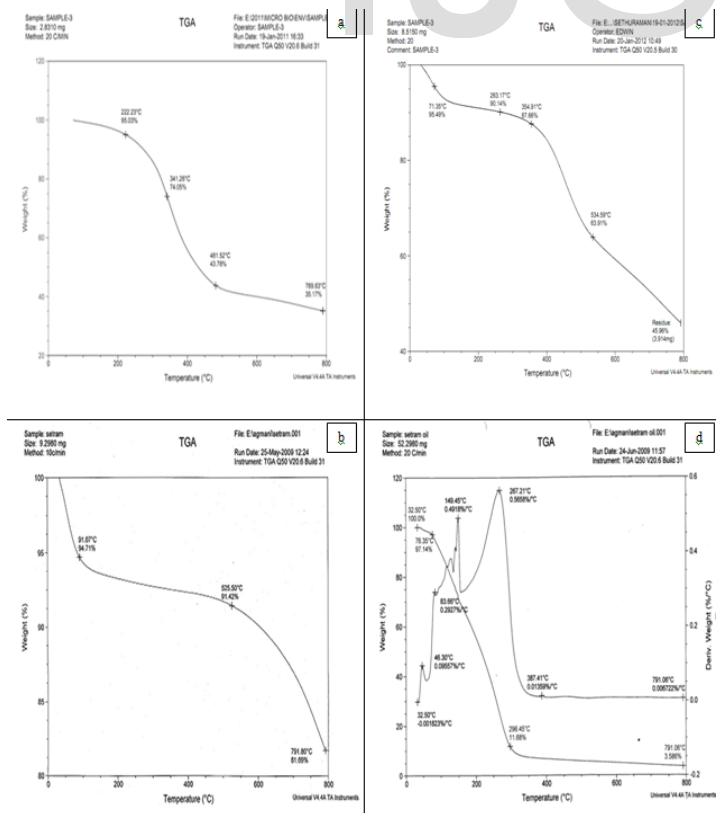


Fig. 4. SEM analysis of a. CTLW, b. residual ash under 1st pyrolysis, c. residual ash under double pyrolysis and d. condensate oil

3.4 Analysis of chromium

Hexavalent chromium (Cr⁶⁺) was analysed as per ISO 17075 by UV-VIS-Spectrophotometer on residual ash obtained from 1st pyrolysis and double pyrolysis and found that the valule was below detectable limit.

The residual ash sample obtained under double pyrolysis process sent for hexavalent chromium (Cr⁶⁺) analysis confirmed its absence, since no peaks at 579 eV was recorded in the XPS analysis as shown in Fig. 5.



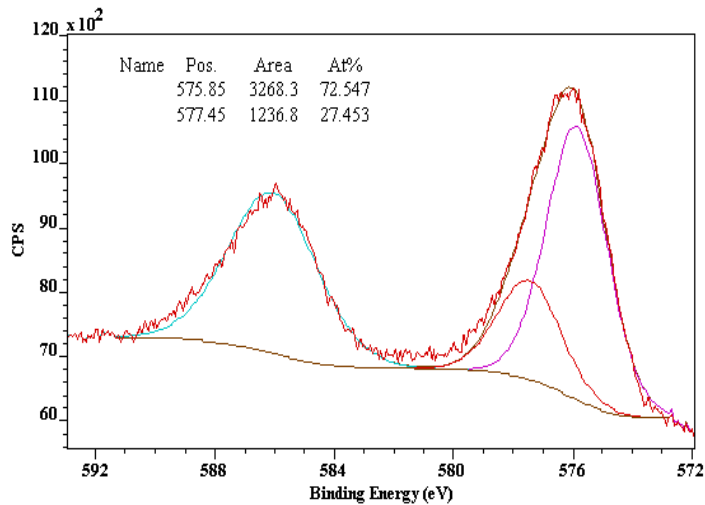


Fig. 5. XPS spectra of residual ash obtained under double pyrolysis

3.5 NMR (¹H, ¹³C) Analysis

In order to study the nature of the protons and carbons in the compounds of the condensate oil, ¹H and ¹³C NMR were taken. Proton NMR spectrum is shown in Fig. 6 and Carbon NMR is shown in Fig. 7. Since, signals are found in wide range from 1 to 8ppm indicates the presence of mixture of compounds rather than single compound. The total no. of protons identified in the spectrum is 77. The downfield shift from 1 to 1.8ppm reveals presence of electro negative atoms in aliphatic mixture. The electronegative atom can be halogen, oxygen or sulphur. Since, there are no signals above 8ppm that shows the absence of oxygen functional groups (i.e., acid and carbonyl groups). The raw material i.e., CTLW used in this pyrolysis process contains no halogen, it can be suggested that the shift has happened due to the presence of sulphur compounds. There are 58 aliphatic protons and 19 aromatic protons.

The number of chemically equivalent carbon atom can be obtained from the C-13 NMR. The signals in C-13 NMR spectrum from 12 to 35ppm indicate the presence of aliphatic hydrocarbon. The observed no signals from 40 to 65ppm reveals the absence of alcoholic and halogen groups. The signals from 110 to 140ppm show the presence of unsaturated hydrocarbons. The signals from 125 to 150ppm show the presence of carbon in aromatic rings. Since, no signals beyond 150ppm show the absence of carboxyl and carbonyl groups in the compounds. The aliphatic and aromatic hydrocarbons attached with sulphur and absence of oxygen enhances the fuel properties and it can also be used as additive for detecting the gas leakage.

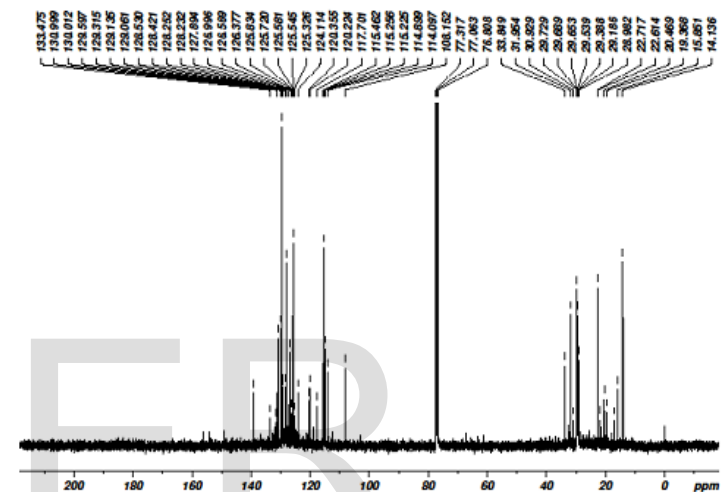


Fig. 7. Carbon NMR (¹³C)

3.6 GC-MS Analysis

The concentration of the major compounds analysed by GC-MS is given in table no. 2.

TABLE 2

CONCENTRATION OF COMPOUNDS IN LIQUID OIL

Name of the compounds	Concentration in mg/L
Benzene	736.87
Ethyl benzene	393.27
4-Isopropylbenzene	29.73
Naphthalene	172.81
Toluene	1376.91
o- Xylene	287.27
m & p- Xylene	199.3

The above table shows that the compound was the mixture of aromatic carbon which increases the fuel property of the liquid oil. Since the number of carbon atoms in the individual compound was not very high, the fuel property of the condensate oil seems to be more. Heavy hydrocarbons are not present in the mixture, if present will suppress the fuel property.

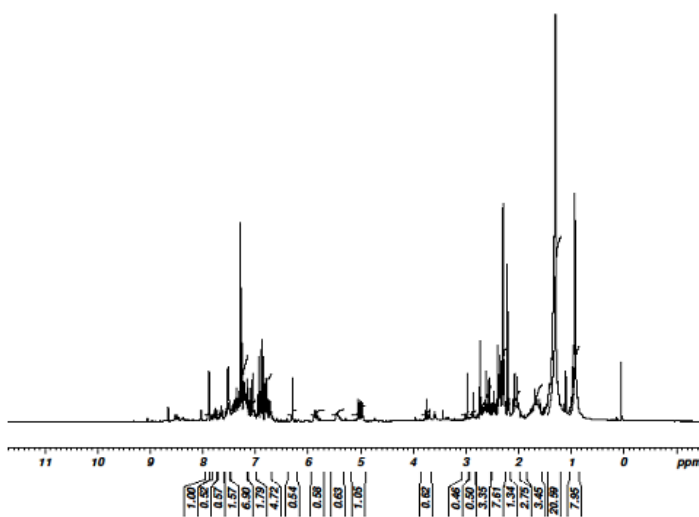


Fig. 6. Proton NMR (¹H)

3.7 Estimation of Ammonium Carbonate

It was found the formation of ammonium carbonate salt when the condensate oil was distilled at 100°C by qualitative method. The compound was found to have ammonium as the cation as the salt was dissolved in water. The anion was found to be carbonate since brisk effervescence was observed when diluted HCl was added to the salt. Hence, the generated salt from the distilled liquid was ammonium carbonate. The digital microscopic image of ammonium is shown in Fig. 8.

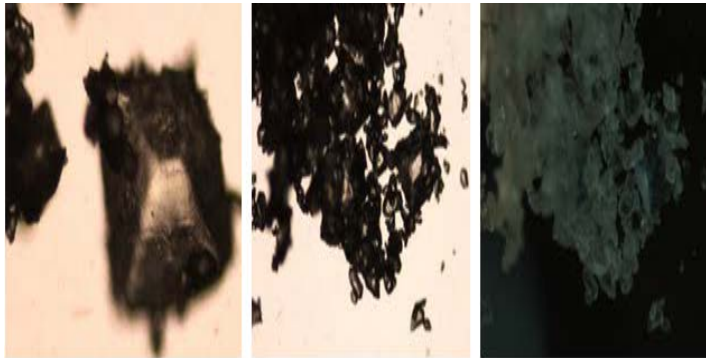


Fig. 8 Images of ammonium carbonate derived from distilled bottom layer liquid of leather solid waste

The following table 3 gives details of mass and balance of the products obtained from pyrolysis and double pyrolysis process.

TABLE 3

MASS AND ENERGY BALANCES OF PRODUCTS RESULTED FROM 1 KG OF CTLW UNDER PYROLYSIS AND DOUBLE PYROLYSIS PROCESSES

Yield	1 st Pyrolysis (I)		Double pyrolysis (II)	
	%	GCV (kJ/kg)	%	GCV (kJ/kg)
Gas fuel ^a	32.01	9529.12	42.45	11490.97
Residual ash	34.67	7865.25	24.23	5903.40
Condensate Liquid fuel ^b	33.32	---	33.32	---
Nitrogen enriched liquid ^c	2.73	380.63	2.73	380.63
Distillate tar	75.24	---	75.25	---
	22.03	---	22.03	---

^a By difference, ^{b,c} Generated out of 333.27g condensate. GCV of residual ash under pyrolysis 22688kJ/kg and under double pyrolysis 24360kJ/kg. GCV of liquid biofuel 41328 kJ/kg.

4. USEFUL PRODUCTS

The clean combustible gas and other useful by-products generated from pyrolysis and double pyrolysis method are shown in the following Fig. 9.

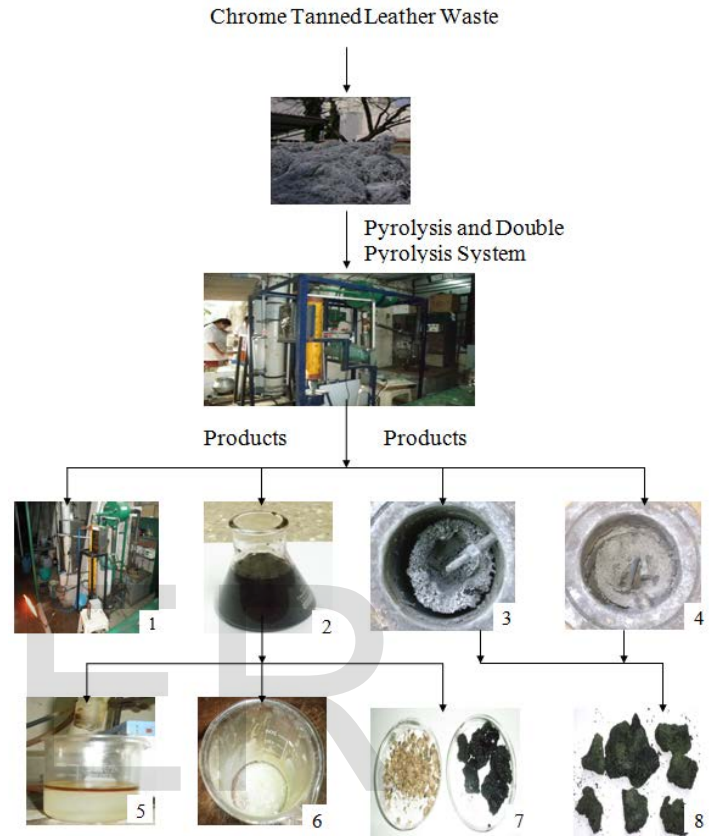


Fig. 9. Useful products derived from hazardous leather solid waste: 1. fuel gas, 2. condensate oil, 3. pyrolysis residual ash 4. double pyrolysis residual ash, 5. distillate condensate oil formed with two layers; top layer is diesel like fuel and bottom layer is nitrogen enriched liquid, 6 ammonium carbonate, 7. remnant distillate tar & residual and 8. carbon residual ash containing trivalent chromium (Cr³⁺)

5. CONCLUSION

The present study is the first kind of attempt on management of chromium containing leather solid wastes through double pyrolysis for its safe disposal while recovering useful products without converting the oxidation state of trivalent chromium (Cr³⁺) into hexavalent chromium (Cr⁶⁺) form in the residual ash. A double pyrolysis process yielded a high energy content combustible renewable gases with minimum (24.23 wt. %) residual mass. The generated gases find wide industrial applications and can considerably replace the thermal energy requirement. The liquid by-products can also find immense applications due to the presence of thiol groups and nitrogen content. The residual carbon ash which contains trivalent chromium (Cr³⁺) can be used in steel manufacturing industries

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