Characterization of Deinking Sludge From Combined Deinking Technology

Kulkarni Shilpa, Chauhan Neetu, Kumar Vivek & Bansal M. C.

Department of Paper Technology, IIT-Roorkee, Saharanpur Campus, Saharanpur - 247 001 (U.P.)

ABSTRACT

The world consumption of recovered paper is estimated at more than 165 million tons, which accounts for more than 40% of the raw material used in papermaking. It is estimated that by 2010, about half of the fibers used in papermaking worldwide will be recycled fibers. Sludge production from the pulp and paper industries has been increasing due to the growth of such facilities. Deinking sludge disposal is one of the most important tasks for environmental protection. Disposal through land filling is expected to decrease due to the limits of existing filling capacities. Alternatives to landfills include the use recycling, composting and incineration, Pyrolysis. Each deinking operation produces sludge with somewhat unique characteristics, depending on the type of waste paper processed.

In the present paper, studies have been carried out on characterization of sludge from old newsprint and Laser printed paper respectively. Sludge of old news print paper recycling is obtained from a mill while sludge from laser printed paper is obtained in the laboratory. Deinking of laser printed paper in laboratory is done by standard chemical deinking process.

Detailed characterisation was carried out to find out moisture content, pH, ultimate and proximate analysis, ash content, inorganic/ organic matter, electrical conductivity, and heavy metal. Thermogravimeteric and differential thermal analysis (TG-DTA) has been carried out to determine the thermal stability. From TGA the reaction kinetics of drying, Pyrolysis and combustion of deinking sludge were also studied. The detail characterization of deinking sludge provides important information for the proper treatment of sludge disposal.

KEYWORDS: *Deinking Sludge, Heavy Metal, Recycling, Ash content, Loss on ignition, Thermal Analysis*

Introduction

The world consumption of recovered paper is estimated at more than 165 million tons, which accounts for more than 40% of the raw material used in paper making **(7).** An increasing proportion of recycled fibers in paper making results in the production of large amounts of de-inking paper sludge (DPS). Removing the ink, clay, coatings and contaminants from waste paper in order to salvage reusable cellulose fibers to produce recycled paper creates deinking sludge which creates disposal problems **(8).** The sludge also creates consumer concern about the potential presence of heavy metals, polychlorinated biphenyls (PCBs) and dioxin **(9).**

Each deinking operation produces sludge with somewhat unique characteristics, depending on the type of waste paper processed and deinking process. Waste paper with high amounts of filler, such as clay-coated magazine paper, produces much lower yields of usable fiber than does waste paper that is mostly cellulose fiber, such as computer printout or old newspaper. Paper that is only lightly printed will produce less sludge than paper that is heavily printed. Alkaline papers contain high filler-to-fiber ratios and, therefore, produce more sludge. Post-consumer feedstock generally produces more sludge than pre-consumer grades because they tend to be more heavily contaminated **(11).** In present scenario different deinking processes are used. Enzymatic deinking is advantageous for industrial usage because it is efficient, quick and has a low environment impact. Besides, UV-irradiation and other technologies have begun to be investigated by some researcher, which is still in its starting stage **(9, 5, 10).**

Characterization of sludge generated from conventional deinking process is well reported in literature. Thermal analysis of deinking sludge from paper industry has been reported by (*Mendez et al*). Characterization of deinking sludge was reported by (*Gea Teresa et al.).*

Recently, landfills are becoming difficult to site and costly to construct and operate because of more stringent regulation, diminishing land availability, and public opposition. The disposal cost of paper sludge is approximately half of the cost of wastewater treatment. Hence, the paper manufacturing industry is interested in reducing the paper sludge disposal cost through utilization of sludge in various applications as shown in **table1**.

Table 1: Utilization and disposal of deinking residues (4)

S.No.	Disposal option	Percentage (%)
1.	Landfilling	5.8
2.	Brickworks	8.1
3.	Cement industry	1.5
4.	Other building materials	6.0
5.	Onsite thermal utilization	65.8
6.	External thermal utilization	12.9
7.	Agriculture	0

Some of research has been carried out on characterization of deinking sludge (physical and chemical) (8).

The present work focuses on the characterisation of deinking sludge generated for various deinking techniques namely i) Conventional deinking, ii) Enzymatic deinking and iii) Combined deinking (UV + Chemical, Enzyme + UV-irradiation). For the purpose, sludge has been generated in the laboratory using photocopier waste paper. Various researchers are investigated on deinking sludge but no result was found on the characterization of combined deinking sludge

Table 2: Pulping condition and chemical dosing

Specification	**Chemical deinking**	Enzymatic deinking
Hydrapulper consistency	10%	10%
Pulping time of hydrapulper	25-30 min	25-30 min
Temperature	60ºC	45ºC
Pulping chemical	Sodium hydroxide- 2.0% Sodium silicate -2.5% Hydrogen peroxide- 1.0% DPTA -0.5%	-
Surfactant	Oleic acid -1%	Oleic acid-1%
Enzyme dosing	-	Cellulase-0.4IU/ml
pH	-	4-5
Enzyme incubation	-	45 min

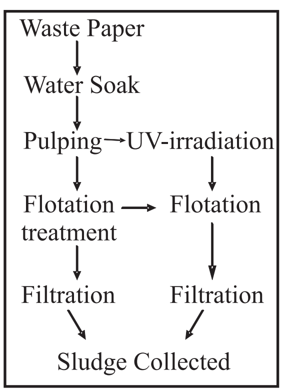
2. Material And Methods

2.1 Raw Material

Photocopier grade paper (basis weight 70 gsm) was used to produce waste paper after printing with Xerox Black toner, using Xerox Workcenter pro420 photocopier machine. All sheets were uniformly printed.

2.2. Experimental Plan

Flow chart of experimental plan is show in figure1 (a) Chemical deinking and combined deinking and figure1 (b) Enzymatic deinking and combined deinking



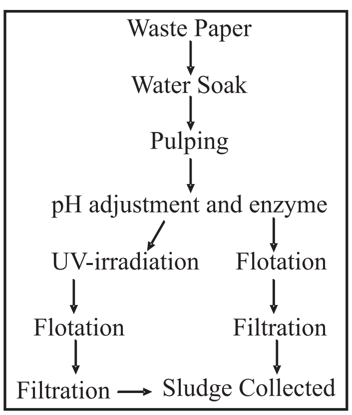


Figure:1(a) Chemical deinking and combined deinking

Figure:1(b) Enzymatic deinking and combined deinking

2.2 Deinking process

2.3.1 Pulping

Hydrapulper is used for slushing of the xerographic paper used during the process under controlled temperature and at fixed rotor speed 180 rpm. The detailed conditions for chemical dosing and enzymatic pulping are shown in **Table 2.**

2.3.2 Flotation cell

In the Flotation stage, about 100 g oven dry repulped stock from the hydrapulper was diluted to 1% consistency and about 10L. Diluted stock was sent to the batch flotation cell. The flotation was performed in the Lambort type flotation cell. The rotor speed was 1800 rpm.

2.3.3 Enzyme

Commercial cellulase enzyme (Hi-media) was used for deinking purpose. The enzyme was stored at 4°C prior to use. This enzyme acts mainly on the surface of the fiber in the acidic to neutral pH range (pH 4.0 to 5.0) and between temperatures of 40 and 50°C. The cellulase activity was measured using the carboxymethyl cellulase (CMCase) method (*Mandels et al. 1976*). Its highest activity was found to be 8.60 IU/mL (0.1g in 10ml) at 50°C and pH 4.8. (3).

2.3.4 UV irradiation treatment

UV irradiation treatment was carried out in a plastic container. The plastic container containing the pulp mixture was placed in a timber-framed UV (ultra violet) reactor (77cm ×36cm × 71cm) equipped with (λ =365nm), 72W (Philips) on the top side, located at a distance of 15cm from the sample. The reaction mixture was subjected to irradiation under UV light with continuous stirring. A fan fitted on the side wall was used to lower the heat generated by UV lamps. All the experiments were carried out in completely mixed and batch mode (**2).**

2.4 Sludge characterisation

Sludge obtained by the flotation of the waste paper were characterised for:

2.4.1 Physical properties

Moisture content, pH, ash content, was measured. Moisture content and ash content can be determine by Tappi test method (T 258 om-11and T413 om-06).

2.4.2 Heavy metal analysis

Heavy metal analysis of the sludge is done by the AAS (atomic absorption spectroscopy GBC Avanta version 1.3) for metals like Zn, Cu, Pb, Fe, and Mn.

2.4.3 Thermal properties

Thermo gravimetric analyses of deinking sludge were carried using thermogravimetric analyzer (TG209 F3 taraus ISO 11358). The thermal analysis was carried out in the temperature range of 25°C to 900°C with the heating rate of 20°C per min under the flow of nitrogen at 40 ml per min (23).

3. Result And Discussion

3.1 Deinking efficiency

The deinking efficiency of the process is evaluated through the means of the brightness measured through the TAPPI standard T452. To quantify this deinking efficiency, the brightness of the hand sheets is compared with the reference sheet. The brightness of the unprinted with all the same chemicals as in the printing paper is considered as reference sheet. The deinkability factor is calculated through the formula given below **(3)**

$DF= (B_F-B_D)/B_{BF}-B_D$

DF=deinkability factor (%)
BF=brightness of pulp in flotation (%ISO)
BD=brightness of pulp after pulping (%ISO)
BBF=brightness of the unprinted paper
The deinkability factor of various processes as shown in **table 3**.

3.2 Sludge Characterization

3.2.1 Physical properties

Moisture, ash content, pH, Yield and loss of ignition of various sludges are presented in **table 3**.
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Result shows that the deinkability factor of the chemical deinking and enzymatic deinking increases for both. Ink removal efficiency of cellulose enzyme is very low (35%) but improved drastically upto 63% with exposure of UV light for 25 min. Effect of UV exposure in chemical deinking is also significant but 9min exposure time seems to be obtained after which improvement in deinking efficiency is not significant.

From the **table 3,** it shows that the deinking efficiency is increasing as the exposure time of the UV is increasing in conventional deinking and after a certain interval of time it become almost constant but in the enzymatic treatment it is first increasing and then decreasing. By analysis the above data shows that 25min is the best time where the removal of the ink is maximum in both the conventional as well as the enzymatic deinking

3.2.2 Heavy Metal analysis

Heavy metal analysis of the sludge is done with the help of the Atomic absorption spectroscopy. Here we are comparing the different metal concentration of the chemical and enzymatic treatment. From **table 4**, it is being observed that the concentration of the metal is present in very less amount in the sludge. Data given below is the concentration of the sludge the highest time that is at 25 min UV treatment still concentration of metal is present in very low amount.

From the heavy metal analysis it is found that there is no significant change in the metal concentration with change of deinking process. It shows that there is no chelating effect of enzymatic treatment and UV exposure

Table 3: Physical properties of sludge

		Chemical sludge						**Enzymatic sludge**					
S..No	Time of UV treatment	Moisture content	pH	D.F.	Yield of sludge	Ash content	Loss on ignition	Moisture content	pH	D.F.	Yield of sludge	Ash content	Loss on ignition
1	0 min	82.6	8.46	62.56	78.83	38.54	45.18	80.0	7.14	35.08	74.32	21.53	31.16
2	3 min	80.0	8.82	65.16	82.51	32.69	39.06	81.4	7.94	39.47	80.21	28.68	33.74
3	6 min	81.0	8.99	74.015	82.30	26.52	37.02	81.3	7.70	39.79	78.97	33.05	
4	9 min	82.1	9.09	79.60	76.73	35.51	27.47	82.3	7.58	28.32	80.21	44.28	44.86
5	25 min	81.2	9.38	79.02	79.44	26.30	38.92	79.9	6.86	63.15	81.92	39.52	49.12
6	45 min	82.4	9.16	81.95	83.07	42.09	44.21	81.0	6.95	53.19	82.14	62.07	69.28

Table 4: Heavy metal analysis metal in the sludge

S.no	**Parameter / Time**	**Conc. of chemical treatment (mg/g)**				**Conc. of the enzymatic treatment (mg/g)**			
		3	6	9	25	3	6	9	25
1	Cu	0.084	0.071	0.063	0.095	0.072	0.061	0.052	0.085
2	Pb	0.045	0.030	0.036	0.311	0.035	0.026	0.029	0.301
3	Ni	0.019	0.014	0.017	0.167	0.016	0.010	0.015	0.081
4	Fe	1.123	1.45	1.76	2.405	1.110	1.30	1.50	1.783
5	Mn	0.48	0.33	0.67	0.583	0.30	0.20	0.54	0.328
6	Zn	0.027	0.028	0.014	0.106	0.01	0.016	0.010	0.141

3.2.3 Thermal characterization`

TGA/DTG curves of chemical and enzymatic deinking with 9 min and without UV treatment and with 9 min UV treatment

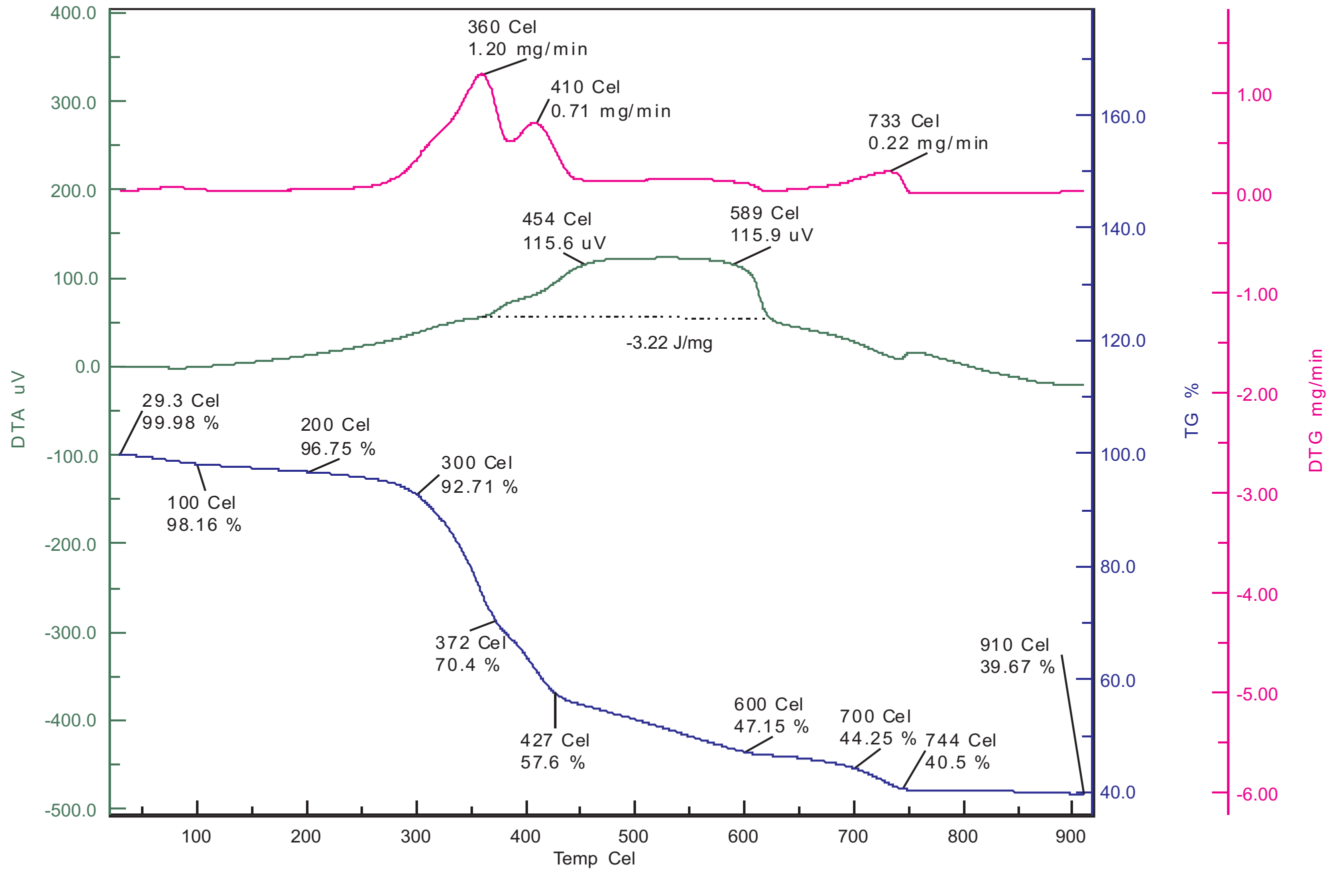

Figure 2: Chemical deinking sludge

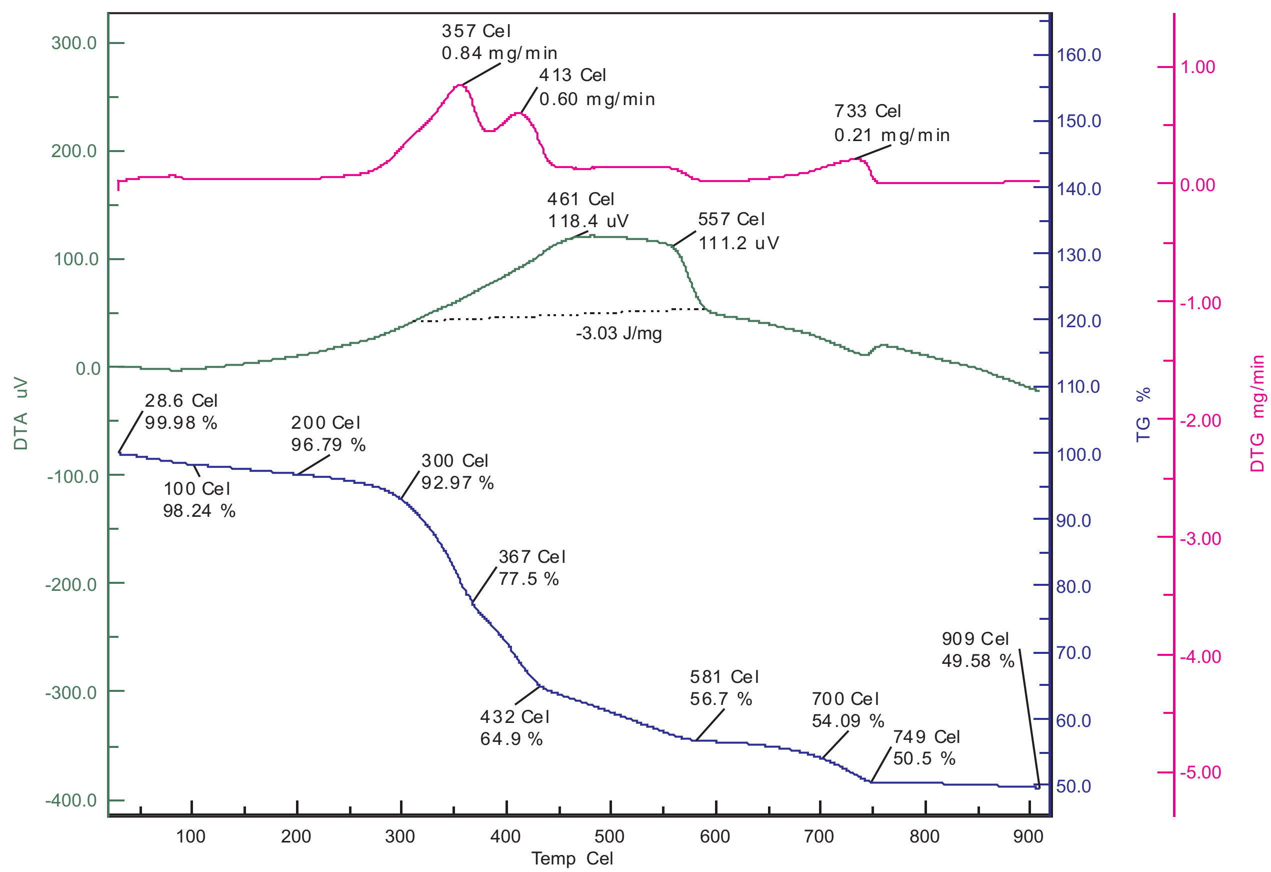

Figure 3: 9min UV Chemical deinking sludge

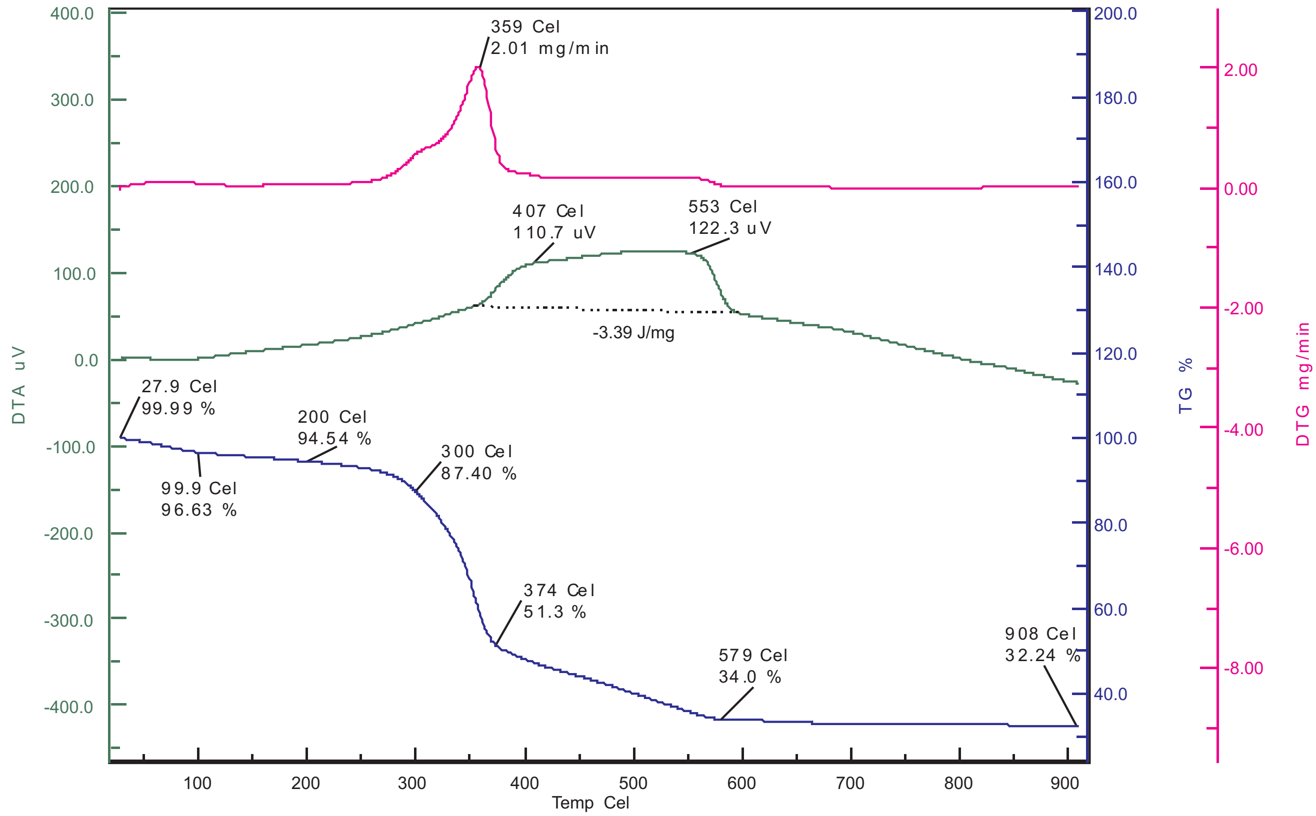

Figure 5: 9 min UV Enzymatic deinking sludge

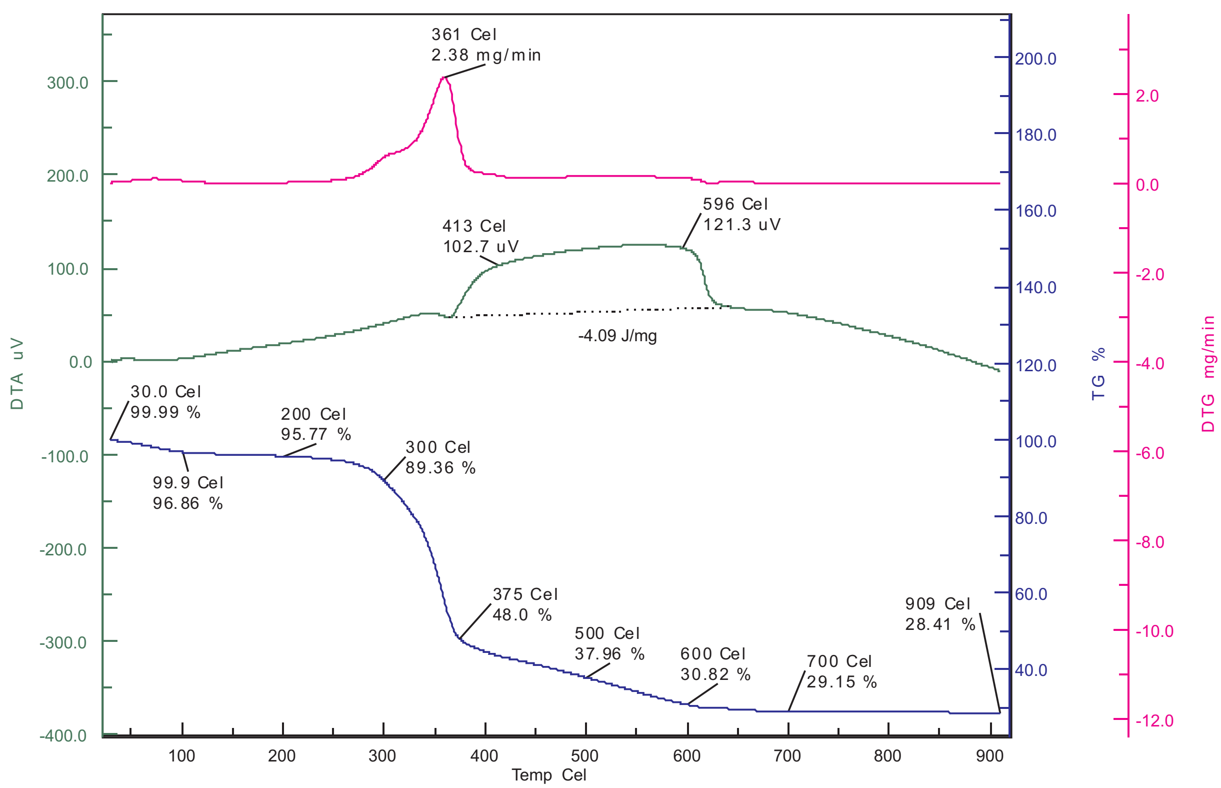

Figure 4: Enzymatic deinking sludge

are presented in Figure (2-5) respectively. Figure 2, 3, 4, and 5 shows chemical only, enzyme only, UV treatment (9min) with chemical and UV treatment (9min) with enzyme . The first weight loss is very small which must be due to the presence of the moisture content in the sludge and it ranges from ambient to 300° C. The second weight loss which is major is due to the presence of the small fibre in deinking sludge and decomposition of the carbon side chain and it ranges between 300ºC -600ºC. The third distinct weight loss is due to the calcinations of the residual carbon species present in the ash and it ranges between 600ºC-900ºC.

Futher ash residues (in percentage), organic to inorganic ratio and loss on ignition of the chemical and enzymatic treated sludges. Further ash %, organic to inogranic ratio and loss of iginition are presented of all types of sludges are presented in **table 5**.

Table 5: Ash and loss in ignition of chemical and enzymatic deinking sludge:

Deinking sludge	Chemical deinking		Enzymatic deinking	
	Ash % in residues	Organic: inorganic ratio	Ash % in residues	Organic :inorganic ratio
Without treatment	39.67	54:46	28.41	70:30
3	36.66	56:44	31.63	68:32
9	49.58	43.6:56.4	32.24	67:33
25	33.42	63:37	41.32	57:43
35	32.94	61:39	47.08	51.7:48.3
45	8.03	60:40	54.18	46:54

The results show that slight decrease in the yield of sludge with exposure of UV for enzymatic deinking which may be due to reduction in fiber loss in the sludge which is also supported by data obtained by oven method and organic to inorganic ratio obtained by TGA curve. In case of deinking sludge there is a decrease then sludge yield increases with increasing UV exposure, this may be due to the fact that greater amount of UV exposure cellose degradation and more fines yield with the sludge. This is also supported to inorganic to organic obtained from. This reduction may be due to reduction of fine in particles due to UV Irradiation.

CONCLUSION

- Result shows that the cellulase enzyme are not as much effective as conventional deinking process in ink removal from photocopy paper (for the deinking of photocopier paper). pulping followed by uv tretment improves ink removal efficiency as well as fibre recovry. however increasing uv exposure time does not improve the process after a certain amount of energy expose.

- Heavy metal concentration is not important. heavy metal is present in sludges is very less quantity. Among all the metals iron % was found to be highest.

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