



Asian Journal of Chemistry; Vol. 23, No. 11 (2011), 5155-5156

# ASIAN JOURNAL OF CHEMISTRY

www.asianjournalofchemistry.co.in



## NOTE

### Identification of the Extractives Chemical Compounds in Newsprint by GC/MS Methods

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(Received: 10 September 2010;

Accepted: 30 July 2011)

AJC-10233

In this work, paper samples were chosen randomly from paper newsprint produced in Mazandaran Pulp and Paper Mill. At first stage, the paper flour provided and then measured extractive per cent by the TAPPI standard methods. The results showed that average of extractives was 6.92 % in newsprint. In the second stage, paper flour washed by toluene-ethyl alcohol with the 2:1 ratio and added BSTFA to extractives residue. Then the samples kept in Ben Marry Bath in 70 °C for 1 h and they were analyzed by GC/MS. Finally, the GC diagram was used for identification of the compounds; it shows the abundance and retention time of each compound and calculation of quartz index and Adams table. Generally, 58 compounds definitely present in newsprint. The benzaldehyde (32.56 %), trimethyl silane (12.65 %), bis(2-ethylhexyl)phthalate (6.98 %),  $\gamma$ -sitosterol (4.84 %), *n*-hexadecanoic acid (4.56 %), dibenzyl (3.82 %) were the main compounds and the dodecanoic acid (0.14 %), decane (0.18 %), xylene (0.19 %), 1-methyl-4-phenylmethyl benzene (0.2 %) were present in low amount, respectively.

**Key Words:** Newsprint, Retention Time, GC/MS.

The newsprint is susceptible to photo-oxidative reactions which cause the pulps to become discoloured and brightness reversion<sup>1</sup>. Andraday and Searle<sup>2</sup> found that newsprint exposed to polychromatic radiation showed the largest amount of yellowing when subjected to irradiation at 330-385 nm (UV-A region), these phenolic and carboxylic news groups formation from quinines radicals during photo-yellowing. Identification extractives have been proposed or considered as initiators or the main cause of yellowing in high-yield and mechanical pulps. Hosseini<sup>3</sup> reported that exhaustive ethanol-toluene of the bark and heartwood of *Juglans regia* L. afforded pale red-coloured extractives 10 and 12 % yields, respectively. Detailed chemical evaluation of these extracts using GC/MS revealed the major components in the bark and heartwood extractives to be the 3,7-dioxa-2,8-disilanonane, 2,2,8,8-tetramethyl (25.17 %), while the major heartwood extractives constituent was benzoic acid, 3,4,5-tri(hydroxyl)/gallic acid (44.57 %). The same components of the bark and the heartwood also contained amounts of the 3,7-dioxa-2,8-disilanonane gallic acid, 2,2,8,8-tetramethyl and 2,3,4,5,6-pentakis-O-(hydroxyl)-d-glucose. The most toxic components in the heartwood, were juglone (5.15 %) and 2,7-dimethylphenantherene (5.81 %). Gupta *et al.*<sup>4</sup> reported that the durability of walnut has been related to the presence of phenolic compounds such as flavonoids, naphthaquinones and hydrolyzable tannins. The

wood and the bark of black walnut have not been found to contain tannins. However, the wood contains appreciable amounts of gallic acid as well as ellagic acid, glucose and a dark violet polymer. Monica *et al.*<sup>5</sup> reported that quinone structures and quinone precursors such as hydroquinones and catechols are important reactions in the photo-yellowing process in acetylated ground wood pulps. Qiu and Ni<sup>6</sup> have reported that the catalytic activity of Mn<sup>2+</sup> and Mn<sup>3+</sup> in hydrogen peroxide decomposing studied by using DTPA as the only stabilizer. It was found that addition of DTPA to a Mn<sup>2+</sup> containing system is more effective than if it is added to a Mn<sup>3+</sup> containing system. To decrease the catalytic of Mn<sup>3+</sup>, sodium borohydride and DTPA under an acidic condition were considered to change Mn<sup>2+</sup> to Mn<sup>3+</sup>. The effect of pH on using DTPA to decrease Mn induced peroxide decomposition is discussed. Vaysi<sup>7</sup> by using GC/MS reported that extractive variation of cypress tree decrease in the longitudinal direction. The average of extractives in natural and planted cypress tree are 7.52 and 2.57 %, respectively. There are specified 14 compounds in natural and 12 compounds in planted cypress tree, *e.g.*, isophyllocladene, 9-octadecenamide, cinnamaldehyde-2-hexyl, bourbonanone and 1*H*-naphtho[2,3-*c*]pyran-3-acetic acid there were in either species so much and these compounds are very important in durability.

In this work, paper samples were chosen randomly from paper newsprint produced in Mazandaran Pulp and Paper Mill. At first, paper flour then measured extractive by TAPPI standards. Then paper flour washed by toluene-ethyl alcohol with the 2:1 ratio. The pure extractives obtained were separated and dried by nitrogen gas to give pale red coloured extracts in 10 and 12 % yields from the newsprint. In order to identification of extract, about 1 mg solid extracts obtained, mixed with 30  $\mu$ L BSTFA + 1 % TMCS reagent and about 15  $\mu$ L pyridine inside tube test. The samples kept in Ben Marry Bath in 70  $^{\circ}$ C for 1 h and they were analyzed by using GC/MS on an HP 6890 gas chromatograph, equipped with a split/split less injector and a 5973 mass selective detector (MSD). The column oven was programmed as follows: chromatography was performed on a HP-5MS capillary column (SGE, 30 m, 0.25 mm), kind of carrier gas, helium with 1 mL/min speed and temperature program between 60-260  $^{\circ}$ C, increase temperature 6  $^{\circ}$ C/min. For the identification of compounds were used GC diagram which shows abundance and retention time of each compound, calculation of quartz index and Adams table.

$$I = 100n + 100(t_{rx} - t_m)/(t_m + 1 - t_m)$$

where: I: quartz index, n: carbon number of normal Alcan,  $t_m$  + 1: retention time of unknown compound,  $t_m$ : retention time of normal Alcan.

The results of this study showed the average of extractives was 6.92 % in newsprint. The 58 compounds were present in newsprint viz., benzaldehyde (32.56 %), trimethyl silane (12.65 %), bis(2-ethylhexyl)-phthalate (6.98 %),  $\gamma$ -sitosterol (4.84 %), n-hexadecanoic acid (4.56 %), dibenzyl (3.82 %) and octadecanoic acid (3.55 %) were main components and dodecanoic acid (0.14 %), decane (0.18 %), xylene (0.19 %), 1-methyl-4-phenylmethyl benzene (0.2 %) and tetradecane (0.37 %) were present in low amount (Fig. 1 and Table-1).

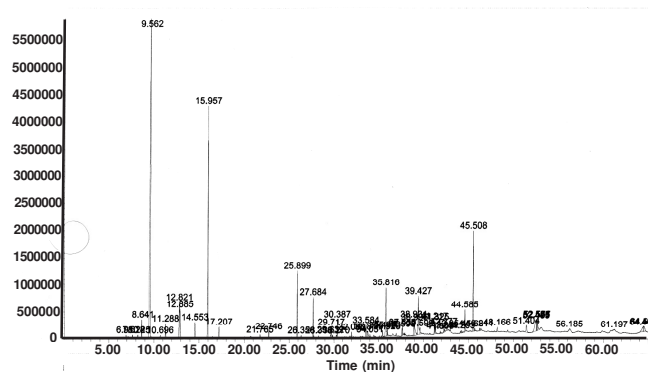


Fig. 1. Gas chromatograph of extractives chemical compounds in newsprint

TABLE-1  
ANALYSIS OF IMPORTANT EXTRACTIVES CHEMICAL  
COMPOUNDS IN NEWSPRINT BY GC/MS

Chemical component	Retention time (min)	Area (%)	KI
Xylene	6.910	0.19	1000
Benzaldehyde	9.562	32.53	1049
Decane	10.694	0.18	1237
Benzyl alcohol	12.823	2.97	1218
Trimethyl silane	15.96	12.65	1336
Dodecane	17.208	0.63	1384
Tetradecane	22.745	0.37	1614
Bibenzyl	25.902	3.82	1759
Propanoic acid	27.68	2.27	1844
Dodecanoic acid	28.315	0.14	1876
Phenyl ethanol	30.385	1.37	1981
Dibutyl phthalate	35.44	0.48	2250
n-Hexadecane	35.819	4.59	2268
Oleic acid	38.995	1.84	2473
Octadecanoic acid	39.428	3.55	2672
bis(2-Ethylhexyl) phthalate	45.5	6.98	4550
$\gamma$ -Sitosterol	52.5	4.84	5250

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