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Discovering the Complex Realm of Semi Volatile Organic Compounds (SVOCs) in Sugarcane Distillery Spentwash Using GC-MS Analysis

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Abstract
Anthropogenic pollutant of sugarcane based distilleries, is one of the major source of environmental pollution like air, water, soil & its disposal is challenging due to pH, colour, solid content, inorganic & organics compounds. The estimation of Basic, Neutral & Acidic SVOCs presents challenges due to the simultaneous measurement of compounds over a wide concentration range. The accelerated solvent extraction (ASE) method involves sample extraction with methylene dichloride & concentrate solvent extracts by using Kuderna-Danish (K-D) concentrator glassware tool. The ASE method is simple, faster, more expedient & cost effective than previously available methods which require tedious sample preparation with complex sample holders. The study allowed obtaining qualitative identification and distribution of base, neutral & acidic SVOCs of Indian sugarcane distillery effluent was studied by using GC-MS. The GC-MS profile of Basic, Neutral & Acidic SVOCs in the range of 100-200 amu are prominently observed in juice, raw and biomethanated spentwash sample. Neutral SVOCs are conspicuous in juice (68.18%) and biomethanated (56.03%) spentwash sample in MW range of 100-500 amu. Acidic SVOCs are prominent in raw (53.45%) spentwash sample in MW range of 100-500 amu. The overall percentage of SVOCs leads to pH of spentwash sample.

The distribution of SVOCs in untreated juice (44 BNAs), raw (111 BNAs) & biomethanated (140 BNAs) samples was estimated by using direct injection GC-MS, whereas the thorough assessment of their possible sources will be the aim of a next investigation.Therefore, determining the qualitative assessment of SVOCs in different stages is one of the important priorities of environmental ecosystem, which is doubly important in terms of its negative effects on environmental health.

Keywords: BNAs (Base, Neutral and Acids), ASE (Accelerated Solvent Extraction), KD (Kuderna-Danish), GC-MS (Gas Chromatography-Mass Spectrometry), JSW (Juice Spent Wash), RSW (Raw Spent Wash), BSW (Biomethanated Spent Wash)

Introduction
Sugarcane is widely used industrially and provides 60-70% of the sugar produced in the world and other valuable products like jaggery, syrup, cane honey and molasses. Sugarcane is still an important food crop in tropical subtropical climatic zones. In 2021, this crop was cultivated by nearly hundred countries and almost 2 billion tons of cane were harvested. The top producers were Brazil (715 million t), India (405 million t) and China (213 million t) [1].

India is second largest sugarcane producer globally after Brazil and contribute substantially to economic development to the rural population. Molasses is a cheap source for production of alcohol in distilleries by fermentation method [2]. The distilleries are one of the largest industries, generating huge quantities of effluent like raw effluent/spent wash, which is potentially a great cause of aquatic and soil pollution [3]. The most damaging effect of the wastewaters of the fermentation industries, especially sugarcane based distillery, on a stream is caused by high concentration of readily decomposable organic matters present in the waste waters. Due to decomposition of soluble and suspended organic matters present in the wastewaters, high BOD (Biochemical Oxygen Demand) and COD (Chemical Oxygen Demand) of the wastewaters results, causing fast depletion of the oxygen content of the water, thus creating a foul smell. This further promotes growth of annoyance organisms and can render the stream totally unhealthy for propagating fish life and for the purpose of drinking, personal hygiene, restitution and other purposes. Water pollution is a concern for the world due to presence of various contaminants and their negative impacts on the ecosystem [3]. Distilleries, pulp and paper industry, oil refineries, pharmaceutical, textile, paint industry, dairy and flour mills etc. are promote for higher water contamination [4]. Sugarcane distillery waste is one of the source of aquatic pollution generating in large volumes. This high strength of waste water containing molasses with disobedient properties. This effluent is dark colour and foul smelling particularly rich in organic matter and lead to acute ecological problem when released untreated into the environment [5-9]. Inadequate and indiscriminate disposal of distillery wastewater deteriorates the quality of the soil, air and groundwater. Water quality is vital for people's well-being and the environment. Contaminated water quality, can lead to numerous adverse health problems.

When the untreated effluents get released into surface water resources, the intense dark brown color hinders the photosynthesis & eutrophication process. Further the acidic pH of the waste causes bioavailability of element and shows impacts on agricultural crops and the overall aquatic microbiome [10-12]. Moreover, the high concentration of COD, BOD and biodegradable organic materials like carbohydrates, lignin, hemicellulose, dextrin and organic acids were also commonly present in the distillery spent wash effluent [13,14]. The physicochemical characterization and organic constituents in distillery spent wash and primary treated distillery effluent was studied by Anusha etal [15]. Most of the compounds in spent wash sample like alcohol, ketones, esters, amino group, peptides and metabolites are estimated by using LC-MS QTOF technique [16]. The main components of distillery effluents are carbohydrates, lipids, proteins, amino acids, fatty acids, organic acids, lignin, melanoidin and their fragmentary products with inorganic metal complexes [17-19]. Odorous substances emitted from industrial sources include both inorganic and organic gases and particulate odorous compounds resulting from biological activity or are present in emissions from chemical processes under normal atmospheric conditions with significant volatility. P.A.Clausen studied emission of volatile and semi-volatile organic compounds from waterborne paints [20]. US Environmental Protection Agency (EPA) methods for semi volatiles analysis involves extracting 1000 ml water sample with methylene chloride using a continuous extraction & distillation by using Kuderna-Danish apparatus [21,22].

SVOCs are those that have a boiling point intermediate between volatile (like alcohols, esters etc.) and non-volatile compounds (larger molecule like heavy metals or complex organic polymers) are present in sugarcane juice, molasses and spent wash samples. The determination of VOC including esters, higher alcohols, aldehydes, methanol and volatile acids is analysed by simple GC-FID. Among the sample pre-concentration methods, purge and trap (P and T) and solid phase micro-extraction (SPME) were the most chosen method for VOCs. Semi Volatile Organic Compound (SVOC) are subgroup of VOCs and normal distillation, solvent extraction, P and T, SPME methods are not efficient for determination of SVOC. The extraction of SVOC compounds from soil, cane juice, molasses spent wash of sugarcane, can be lengthy and tedious process [23]. Previously, the extraction of environmental materials such as soils, sludge and other solid or liquid wastes required large amounts of solvents. Soxhlet, for example, uses from 250 to 500 mL of solvent for most environmental samples. So accelerated solvent extraction (ASE) method is used for extraction. This is a method for extraction of water insoluble or slightly water soluble volatile and semi-volatile compounds.

Organic compounds have an assortment of direct and indirect effects on human health and on environment through toxicity like carcinogenicity and other adverse effects like photochemical oxidation & reduction.

A literature review has suggested a disposal process based on analyzing different parameters is mentioned in table 1 [24-51]. This table offers essential insights into the work conducted on spentwash. Most research publications emphasize routine parameters, aligning with the objectives of the respective work. This review of previous work and the understanding of sw handling form the foundation of this manuscript. Surprisingly, little attention has been given in the scientific community to developing a specific treatment method for the presence of semi-volatile compounds in the sw. Therefore, we have decided to focus on developing methods to extract semi-volatiles and analyse them using GC-MS. The current work is only to showcase the effective method of extraction of semi-volatile compounds and their qualitative assessments. Further, the quantification of specific groups of semi-volatile compounds is underway.

Author(s)	Article Title	Theme of the Article	Key Findings
[24]	Synthesis of MoS_2 nanomaterials for photodegradation of spent wash	To create and analyze nanoparticles based on MoS_2 , specifically MoS_2/TiO_2, degrading spent wash by photodegradation	Catalyst has good photocatalytic performance and good biocompatibility
[25]	Degradation of Spent Wash Colour: A Review of Treatment Methods	A Review of treatment method transition from a linear waste management approach to a circular economy model	Comprehensive overview of the various treatment methods employed for DSW decolorization
[26]	Biogas Production through Anaerobic Codigestion of Distillery Wastewater Sludge and Disposable Spent Yeast	Biogas Production	Biogas Production through Anaerobic Co-digestion method
[27]	Composting of distillery spent wash	Use of spentwash for composting	Use of SW in Composting.
[28]	Arbuscular mycorrhizas accelerate the degradation of colour containing organic pollutants present in distillery spent wash leachates	Reduction of colour	Colour containing organic pollutants present in distillery spent wash leachates
[29]	Detection and identification of hazardous organic pollutants from distillery wastewater by GC-MS analysis and its phytotoxicity and genotoxicity evaluation by using Allium cepa and Cicer arietinum L	Detection of hazardous organic pollutants by GC-MS	Detection and identification of hazardous organic contaminant in distillery waste water
[30]	Aerobic composting of sugar pressmud with stabilized spentwash and selected microbial consortium	Microbial study using aerobic condition on spentwash	Effect on EC, TS, BOD, COD, VS, Chlorides, Sulphatees
[31]	Decolourization of Distillery Spent Wash by Bacillus coagulans.	Microbial decolourization of distillery spentwash	Colour reduction by microbial method
[32]	Comparative adsorptive performance of adsorbents developed from sugar industrial wastes for the removal of melanoidin pigment from molasses distillery spent wash	Removal of melanoidin pigment from molasses distillery spent wash	Melanoidin pigment removal from molasses based distillery spent wash
[33]	Distillery Stillage: Characteristics, Treatment, and Valorization	Study of distillery stillage & its treatment	Characteristics, Treatment, and Valorization study of distillery stillage.
[34]	Distillery spent wash: An emerging chemical pool for next generation sustainable distilleries	EC, pH, COD, BOD, TDS, TS, TSS, ash, xylose, glucose, sucrose, formic acid, acetic acid, propionic acid, butyric acid	Physico-chemical Parameter studied for sustainable distilleries
[35]	Distillery spent wash (DSW) treatment methodologies and challenges with special reference to incineration: An overview.	Color, pH, smell, COD, BOD, Total N_2, ammonical N_2, P, K, TS, VS, Ash	Impact of incineration method on distillery spentwash
[36]	Sugarcane biorefineries wastewater: bioremediation technologies for environmental sustainability	pH, EC, BOD, COD, Chloride, total hardness, TS, TDS, TSS, Nitrates, Organic N_2, total N_2, phosphate, sulfate, oil and grease	Study of bioremediation technique on sugarcane wastewater
[37]	Photodegradation of spent wash, a sugar industry waste, using vanadium-doped TiO_2 nanoparticles	Photodegradation of spent wash by Metal TiO_2 nanoparticles	Colour degradation using metal doped TiO_2 by Photocatalysis.
[38]	Anaerobic treatment of blended sugar industry and ethanol distillery wastewater through biphasic high rate reactor	Anaerobic treatment of blended sugar industry and ethanol distillery wastewater	Anaerobic treatment of wastewater of ethanol distillery.
[39]	An influence of experimental parameters in the treatment of anaerobically treated distillery spent wash by using ozone assisted electrocoagulation	Anaerobically treated distillery spent wash by using ozone assisted electrocoagulation	Effect of ozone assisted electrogulation on spentwash.
[40]	Decolourization of spentwash: a comparative analysis of physical, chemical and biological approaches	Spentwash decolourization study with physical, chemical and biological approaches	Reduction in colour of spentwash

[41]	Treatment processes and technologies for decolourization and COD removal of distillery spent wash: a review	pH, BOD, COD, TS, TSS, TDS, chlorides, sulphate, phosphates, phenols & colour removal	Physico-chemical Parameter study.
[42]	Decolourization of distillery spent wash effluent by electro oxidation (EC and EF) and Fenton processes: a comparative study	Comparative study of spentwash effluent decolourization by oxidation methods	Reduction in colour of spentwash by electro oxidation
[43]	Decolourization of distillery spent wash using biopolymer synthesized by Pseudomonas aeruginosa isolated from tannery effluent	Decolourization of Distillery Spent Wash Using pseudomonas aeruginosa	Microbial decolourization of spentwash.
[44]	Spent wash decolourization using nano-Al2O3/kaolin photocatalyst: Taguchi and ANN approach	Spent wash decolourization by using photocatalyst	Photocatalytic decolourization of spentwash.
[45]	Bioremediation and Decolourisation of Biomethanated Distillery Spent Wash	Decolourisation of Biomethanated Distillery Spent Wash	Reduction of biomethanated spentwash.
[46]	Detection of persistent organic compounds from biomethanated distillery spent wash (BMDS) and their degradation by manganese peroxidase and laccase producing bacterial strains	Detection of persistent organic compounds from biomethanated distillery spent wash (BMDS)	Detection of Organic compounds in Biomethanated spentwash.
[47]	Removal of colour of spent wash by activated charcoal adsorption and electrocoagulation	Removal of spent wash colour by activated charcoal	Removal of colour by using activated charcoal.
[48]	Polishing of biomethanated spent wash (primary treated) in constructed wetland: a bench scale study	pH, EC, BOD, COD, colour removal	Physico-chemical Parameter study.
[49]	Characterization of spent wash from different distilleries operating in	pH, EC, BOD, COD,TS, TDS, heavy metals, nutrient elemental	Physico-chemical Parameter study.
	Haryana and its utilization as a source of liquid manure in agriculture	composition, VS, SS, Ash, % colour removal	
[50]	Effect of sugar factory distillery spent wash (DSW) on the growth pattern of sugarcane (Saccharum officinarum) crop.	pH, EC, BOD, COD, colour removal, TS, TDS, VS, Ash, % carbonates, bicarbonates, nutrient elemental analysis, heavy metals	Physico-chemical Parameter study.
[51]	Treatment and disposal of distillery spentwash	Colour decolourisation, biogas, total N_2 and K	Different treatment & disposal methods of spentwash.

Table 1: Various Parameters Studied by Authors for Spentwash Samples

Materials and Methods

Materials and Reagents

The chemicals and raw materials were used as received without further treatment/purification. All chemicals are AR grade of Merck make like sodium sulphate anhydrous, dichloromethane, sulfuric acid (68%), sodium hydroxide pellets and pH meter (Metrohm). The water used has conductivity is <0.06 uS. Glassware's like separating funnel, KD concentrator tube, distillation column must be scrupulously cleaned. All materials used in the analysis must be demonstrated to be free from contamination and interferences. A samples of spentwash was brought from cane sugar factory, Pune, Maharashtra, India, in sterile sample bottle and it was directly kept in the refrigeration at 4^0C.

Method

The JSW, RSW and BSW samples were collected for this study were analysed for semi-volatile organic compounds. A measured volume (50 mL) of sample is three time serially extracted with methylene chloride (50 mL) at sample pH followed by basic pH of aqueous phase (0.1 M NaOH) and at acidic pH (0.1 M H_2SO_4) using liquid/liquid solvent extractor. Combine all the organic fractions of sample pH, basic & acidic pH samples and processed further to distillation and concentration. The extraction process was carried out at ambient temperature under the fume hood.

Distillation and Concentration

All three organic fractions were added with 2-3 grams of anhydrous sodium sulphate to remove the moisture if any. Then all the contents from the flasks are carefully transferred to distillation unit which contains K-D concentrator unit. The Kuderna-Danish (K-D) concentrator is a piece of glassware used for concentrating extract in water bath (~50 to 60^0C) to reduce the volume to 1 mL. The concentrate solvent extract of SVOCs of JSW, RSW and BSW samples were subjected for the further GC-MS analysis.

Analytical Technique

The KD concentrated sample analysis was performed with GC-MS instrument (Agilent Technologies) with scanning range from 35-450 amu of every two seconds or less, utilizing a 70 eV energy of EI mode. The instrumental verification was checked with inbuilt PFTBA standards and tune performance done with 50 ng/uL of certified standard of Decafluorotriphenyl phosphine (DFTPP) by auto injector technique. The TIC chromatogram of each sample was verified with NIST search (version 2.0) library. The instrumental analytical conditions are mentioned in table 2.

Gas Chromatograph (GC) Condition Parameters	
Model	Agilent 7890 A Gas Chromatograph
Column Pneumatics	Constant flow, Helium carrier gas
Injector Mode	Split (1:10)
Injector Liner	Inlet liner, split, double tapered deactivated
Injection Volume	2 .0 uL
Injector Temp.	260^0 C
Carrier Flow	0.7 mL/min
Temperature Program	30^0 C for 2 min, 8^0 C/min to 280^0 C, 2 min hold
Equilibration Time	0.10 min
MSD Detector Parameters	
Model	Agilent 5975C Triple Axis Detector
Ionization Mode	EI, 70 eV
Acquisition Mode Auxiliary Temperature	Scan 250^0 C
Ion Source Temperature	150^0 C
Quadrupole Temperature	230^0 C

Table 2: Instrumental Analytical Condition for Estimation of SVOCs

Spentwash Generation from Indian Sugarcane Industry

High demand of ethyl alcohol in various industries with cost effective source like sugarcane is very widespread from many years. The generation of sw at different steps in sugarcane distillery industry are shown in Figure 1.

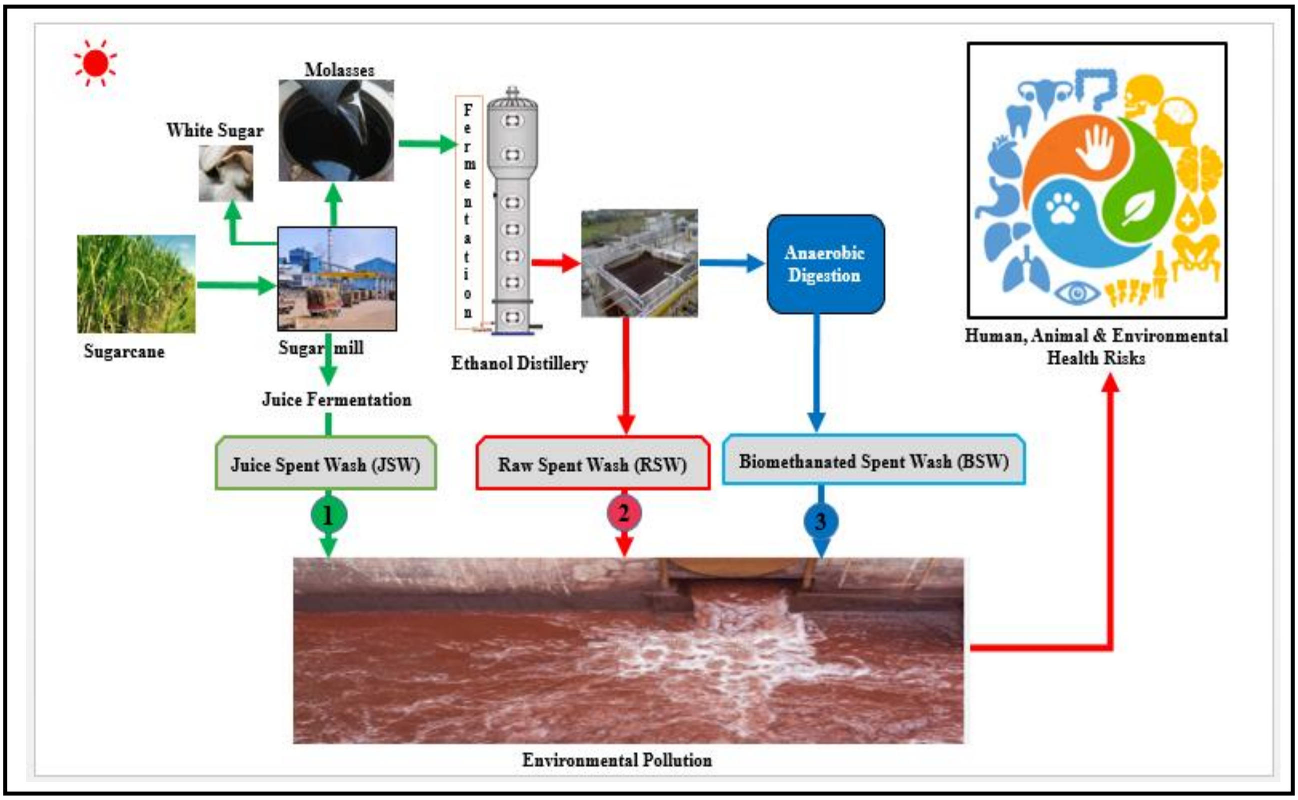

Figure 1: Environmental Pollution & Health Risks by Spent Wash from Sugarcane Based Distillery of JSW, RSW & BSW Sample

Distillery of JSW, RSW & BSW Sample

The process of making ethanol from sugarcane starts when cane stalks are crushed to extract a sugar-rich cane juice and delivered to a fermentation tank where the yeast fermentation reaction occurs to generate ethanol. After distillation, remaining broth is juice sw which contains pH in between 4.0 to 4.5. The cane juice is further treated and concentrated to produce crystalline white sugar and generated dark black/brown coloured viscous waste like molasses which is mainly used for production of ethyl alcohol by fermentation route by using yeast. After distillation residual liquid is dark coloured known as raw sw which has pH in between 3.5 to 4.0. The anaerobic digestion treatment on RSW generated BSW having black-brown colour with slightly smell like molasses which has pH in between 7.8 to 8.0.

Results and Discussion

Semi Volatile Compounds of JSW Sample

The obtained chromatogram demonstrates good separation of analytes. Direct injection of K-D concentrated sample to GCMS has shown a considerable response as observed in TIC chromatogram of JSW sample is presented in Figure 2-A. The sample analysis data was measured for 50 minutes and most of the prominent peak of dichloroacetic acid, were shows high signal intensities than other compounds like 1-propanol, 2,3 butanediol, 1,3 propanediol diacetate, 2-tridecane, Octadecanoic acid ethyl ester, Squalene etc. When juice ferments directly, complex compounds like lipids, waxes phenolics, flavonoids, carbohydrates changes in to simpler forms.

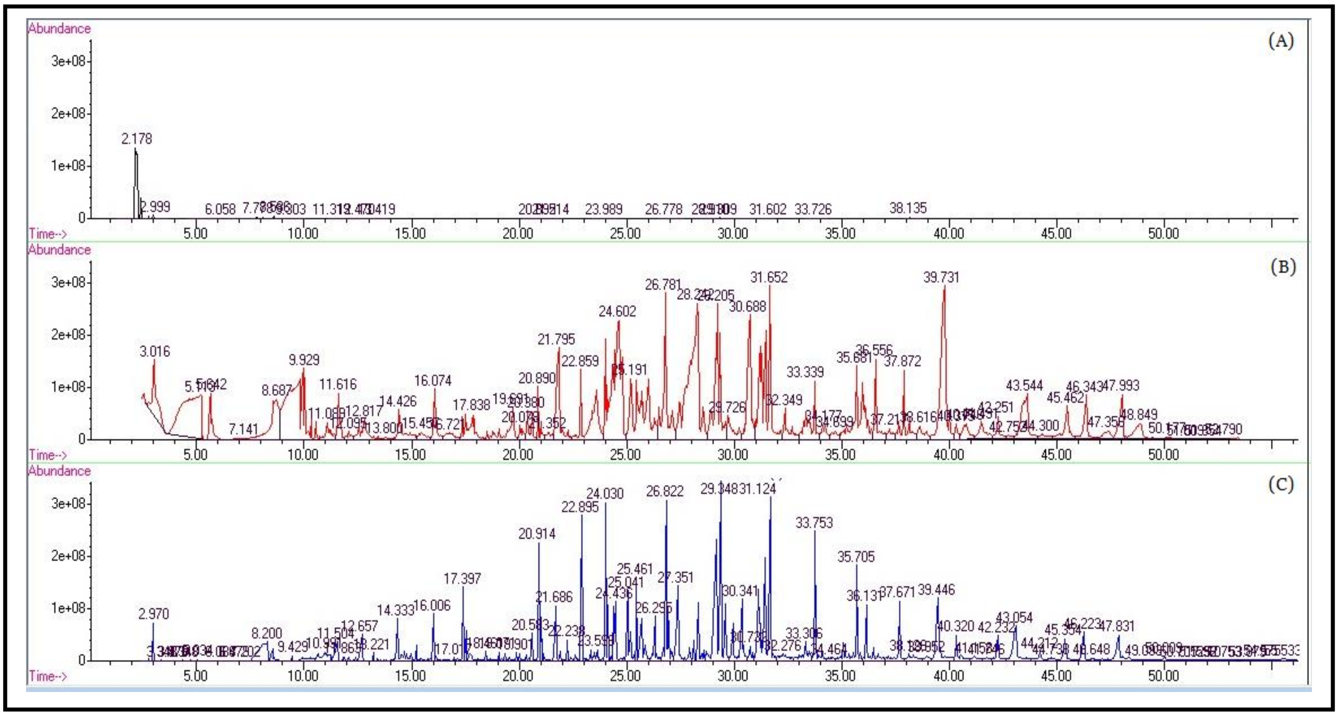

Figure 2: TIC Overlay Chromatogram of SVOCs of (A) Juice Spentwash, (B) Raw Spentwash & (C) Biomethanated Spentwash Sample

All identified compounds by NIST library were categorized into the different groups like aliphatic hydrocarbons, aromatic hydrocarbons, terpenes, sulphur compounds, ethers, alcohols, chloride compounds, esters, carbonyls, nitrogen-containing compounds, and non-identified compounds. Forty-four BNAs compounds were detected in JSW sample (Table 3). The majority of compounds are acid and ester in nature followed by paraffins and olefins compounds. The smell of JSW is ethereal fruity due to ester and alcohol group of compounds. Most of the neutral compounds are alcoholic are produced during fermentation process or cane stale. The compounds in JSW sample was qualitatively identified by using NIST library and are categorised in to their fundamental behaviour like Base (B), Neutral (N) and Acid (A).

RT, minute	Name of compound	MW/ amu	Cas Number	Type of Compound	JSW	RSW	BSW
2.178	Dichloracetic acid	128	79-43-6	A	Detected	Not Detected	Not Detected
2.418	1-Propanol	60	71-23-8	N	Detected	Not Detected	Not Detected
2.649	Acetic acid ethenyl ester	86	108-05-4	N	Not Detected	Not Detected	Detected
2.779	2-Butanol (secondary Alcohol)	74	78-92-2	N	Not Detected	Not Detected	Detected
2.799	sec-Butyl Alcohol	74	14898-79-4	N	Detected	Not Detected	Not Detected
2.918	1,4-Pentanediol	104	626-95-9	N	Not Detected	Not Detected	Detected
2.971	Ethyl Acetate	88	141-78-6	N	Detected	Detected	Detected
3.169	Isobutyl alcohol	74	78-83-1	N	Not Detected	Not Detected	Detected
3.337	2-Methyl-2-butanol-(tert-amyl alcohol)	88	75-85-4	N	Not Detected	Not Detected	Detected

4.286	N-(5-Methyl-3-isoxazolyl) acetamide	140	13223-74-0	B	Not Detected	Not Detected	Detected
4.344	Methyl propenyl carbinol	86	1569-50-2	N	Not Detected	Not Detected	Detected
4.652	Propanedioic acid	104	141-82-2	A	Not Detected	Detected	Not Detected
4.734	2-Pentanol	88	6032-29-7	N	Not Detected	Not Detected	Detected
4.806	Methyl isopropenyl carbinol	86	10473-14-0	N	Not Detected	Not Detected	Detected
4.965	6-Methoxy-2-hexanone	130	29006-00-6	N	Not Detected	Not Detected	Detected
5.018	3-Hydroxy-2-butanone	88	513-86-0	N	Not Detected	Not Detected	Detected
5.223	Propanoic acid, ethyl ester	102	105-37-3	N	Detected	Not Detected	Not Detected
5.278	Methyl propenyl carbinol	86	1569-50-2	N	Not Detected	Detected	Not Detected
5.318	n-Propyl acetate	102	109-60-4	N	Detected	Detected	Not Detected
5.649	1-Propoxy-2-propanol	118	1569-01-3	N	Not Detected	Detected	Not Detected
5.812	3-Isopentenyl alcohol	86	763-32-6	N	Not Detected	Not Detected	Detected
5.991	Isoamyl alcohol	88	123-51-3	N	Detected	Not Detected	Detected
6.101	2-Hexanol	102	626-93-7	N	Not Detected	Not Detected	Detected
6.669	(5-Methyltetrahydro-2-furanyl)methanol	116	54774-28-6	N	Not Detected	Not Detected	Detected
6.718	Isocaproic acid	116	646-07-1	A	Not Detected	Detected	Not Detected
7.052	sec-Butyl nitrite	103	924-43-6	A	Not Detected	Not Detected	Detected
7.146	6-Hydroxy-2-hexanone	116	21856-89-3	N	Not Detected	Not Detected	Detected
7.252	Propylene Glycol	76	57-55-6	N	Not Detected	Not Detected	Detected
7.377	Methyl propenyl carbinol	86	1569-50-2	N	Not Detected	Not Detected	Detected
7.807	2,3-Butanediol	90	19132-06-0	N	Detected	Not Detected	Detected
8.062	Dimethylethylene glycol	90	513-85-9	N	Detected	Not Detected	Not Detected
8.283	2,3-Butanediol, [R-(R*,R*)]	90	24347-58-8	N	Not Detected	Not Detected	Detected
8.576	2-Nitroethyl propionate	147	5390-28-3	N	Not Detected	Detected	Not Detected
8.587	Ethyl 2-hydroxypropanoate	118	687-47-8	A	Detected	Not Detected	Detected
8.740	4-Hydroxy-4-methyl-2-butanone	102	4161-60-8	N	Not Detected	Not Detected	Detected
8.822	Methyl dimethylcarbamate	103	7541-16-4	B	Not Detected	Not Detected	Detected
9.303	1,3-Propanediol	76	504-63-2	N	Detected	Not Detected	Not Detected
9.332	Propionic acid	74	79-09-4	A	Not Detected	Detected	Not Detected
9.429	3-Ethoxy-1-propanol	104	111-35-3	N	Not Detected	Not Detected	Detected
9.944	1,3-Propanediol	76	504-63-2	N	Not Detected	Not Detected	Detected
9.968	3-Nitro-2-butanol	119	6270-16-2	A	Not Detected	Detected	Not Detected
10.011	Propanal dimethyl acetal	104	09/10/4744	N	Not Detected	Detected	Not Detected
10.026	1,3-Butanediol	90	107-88-0	N	Not Detected	Detected	Not Detected
10.329	3-Ethoxy-1-propanol	104	111-35-3	N	Not Detected	Detected	Not Detected
10.531	Furfuryl alcohol	98	98-00-0	N	Not Detected	Detected	Not Detected
10.599	2,4-Pentadienoic acid	98	626-99-3	A	Not Detected	Detected	Not Detected
10.652	Ethyl glycidyl ether	102	09/11/4016	N	Not Detected	Detected	Detected
10.657	3-Methyloxirane-2-carboxylic acid	102	2443-40-5	A	Not Detected	Detected	Not Detected
10.724	Dimethyl Sulfoxide	78	67-68-5	A	Not Detected	Detected	Not Detected
10.936	1,3-Propanediol	76	504-63-2	N	Not Detected	Detected	Not Detected
10.979	Methyl glycidyl ether	88	930-37-0	N	Not Detected	Not Detected	Detected
11.082	Propyl glycidyl ether	116	3126-95-2	N	Not Detected	Detected	Not Detected
11.278	Isopropyl Alcohol	60	67-63-0	N	Detected	Not Detected	Not Detected
11.317	Butyrolactone	86	96-48-0	N	Detected	Not Detected	Detected
11.331	4-Penten-2-ol	86	625-31-0	N	Not Detected	Not Detected	Detected
11.622	Isopropyl glycidyl ether	116	4016-14-2	N	Not Detected	Detected	Not Detected
11.668	Methanesulfonylacetic acid	138	2516-97-4	A	Not Detected	Not Detected	Detected
11.721	Butyrolactone / Butyric acid lactone	86	96-48-0	A	Not Detected	Detected	Not Detected
11.779	Methyl glyoxal	72	78-98-8	B	Detected	Not Detected	Not Detected
11.783	2-Hydroxy-3-pentanone	102	5704-20-1	N	Not Detected	Detected	Not Detected
11.807	2,5-Hexanedione	114	110-13-4	N	Not Detected	Not Detected	Detected
11.884	Propyl nitrite	89	543-67-9	B	Not Detected	Not Detected	Detected
11.947	2,3-Dihydroxypropanal	90	497-09-6	N	Not Detected	Detected	Not Detected
11.957	Butanoic acid, 3-hydroxy-, ethyl ester	132	5405-41-4	A	Not Detected	Not Detected	Detected
12.048	1-Propoxy-2-propanol	118	1569-01-3	N	Not Detected	Detected	Not Detected
12.082	Ethylene glycol monoallyl ether	102	111-45-5	N	Not Detected	Not Detected	Detected
12.092	1-Isopropoxy-2-propanol	118	3944-36-3	N	Not Detected	Detected	Not Detected
12.299	2-Methylbutanolide	100	1679-47-6	N	Not Detected	Not Detected	Detected
12.409	4-Pentanolide	100	108-29-2	N	Not Detected	Not Detected	Detected
12.463	1,3-Propanediol, diacetate	160	628-66-0	A	Detected	Not Detected	Not Detected
12.554	2,5-Dimethyltetrahydrofuran	100	1003-38-9	N	Not Detected	Detected	Not Detected
12.693	4-Hydroxy-2-butanone	88	590-90-9	B	Not Detected	Detected	Detected
13.218	1-Decene	140	872-05-9	B	Not Detected	Not Detected	Detected
13.238	2-Decanol	158	1120-06-5	N	Not Detected	Detected	Not Detected
13.224	1-Decene	140	872-05-9	B	Detected	Not Detected	Not Detected

13.411	Decane	142	124-18-5	A	Detected	Detected	Detected
13.546	Trimethylpyrazine	122	14667-55	B	Not Detected	Not Detected	Detected
13.952	sec-Butyl nitrite	103	924-43-6	A	Not Detected	Detected	Not Detected
14.181	2-Methyl-4-heptanol	130	21570-35-4	N	Not Detected	Detected	Not Detected
14.253	Benzyl alcohol	108	100-51-6	A	Detected	Detected	Detected
14.668	Pantolactone	130	599-04-2	N	Not Detected	Detected	Not Detected
14.672	Propanoic acid, propyl ester	116	106-36-5	N	Not Detected	Not Detected	Detected
14.778	Isovaleric acid, ethyl ester	130	108-64-5	N	Not Detected	Not Detected	Detected
14.926	3-Nitro-2-butanol	119	6270-16-2	A	Not Detected	Detected	Not Detected
14.981	2-Acetylpyrrole	109	1072-83-9	B	Not Detected	Not Detected	Detected
15.159	Methyl pyrrol-2-yl ketone	109	1072-83-9	B	Not Detected	Detected	Not Detected
15.217	1-Hydroxy-3-methylbenzene	108	108-39-4	A	Not Detected	Not Detected	Detected
15.356	2,3-Dihydroxypropanal	90	497-09-6	N	Not Detected	Detected	Not Detected
15.376	Nitrobenzene	123	98-95-3	A	Not Detected	Not Detected	Detected
15.911	Phenylethyl Alcohol	122	60-12-8	A	Detected	Detected	Not Detected
16.132	Isophorone	138	78-59-1	N	Not Detected	Not Detected	Detected
16.324	4-Hydroxycyclohexanone	114	13482-22-9	A	Not Detected	Not Detected	Detected
16.676	3-(2-Methyl-1,3-dioxolan-2-yl)-1-propanamine	145	66442-97-5	B	Not Detected	Not Detected	Detected
16.993	3-Ethylphenol	122	620-17-7	A	Not Detected	Not Detected	Detected
17.148	Octanoic acid	144	124-07-2	A	Not Detected	Not Detected	Detected
17.365	1-Dodecene	168	112-41-4	N	Detected	Detected	Detected
17.427	Butoxyethoxyethanol	162	112-34-5	N	Not Detected	Not Detected	Detected
17.499	Creosol	138	93-51-6	A	Not Detected	Not Detected	Detected
17.509	trans-2-Hexenyl benzoate	204	76841-70-8	N	Not Detected	Detected	Not Detected
17.519	Tridecane	184	629-50-5	N	Detected	Not Detected	Not Detected
17.528	Dodecane	170	112-40-3	N	Not Detected	Not Detected	Detected
18.452	trans-4,5-Epoxydecane	156	56740-10-4	N	Not Detected	Not Detected	Detected
18.505	2-Methyl-3-propylpyrazine	136	15986-80-8	B	Not Detected	Not Detected	Detected
18.512	1-Nitro-2-octanone	173	16067-01-9	A	Not Detected	Detected	Not Detected
18.655	Hexanoic acid, 2-phenylethyl ester	220	6290-37-5	N	Not Detected	Detected	Not Detected
18.664	Phenol, 2-propyl	136	644-35-9	A	Not Detected	Not Detected	Detected
18.775	5-Ethyl-2-methyl-pyridin-4-amine	136	5350-64-1	B	Not Detected	Detected	Not Detected
18.828	Phenylmalonic acid	180	2613-89-0	A	Not Detected	Detected	Not Detected
18.852	Methyl[4-(1-pyrrolidinyl)-2-butynyl] formamide	180	18327-40-7	B	Not Detected	Detected	Not Detected
18.997	Salicin	286	138-52-3	N	Not Detected	Not Detected	Detected
19.064	4-Hydroxy-3-methoxy ethyl benzene	152	2785-89-9	A	Not Detected	Not Detected	Detected
19.16	Sulfurol	143	137-00-8	B	Not Detected	Not Detected	Detected
19.339	2-Methyl-4-propyloxetane	114	7045-79-6	B	Not Detected	Not Detected	Detected
19.469	2-Nonen-1-ol	142	22104-79-6	N	Not Detected	Not Detected	Detected
19.536	3-(2-Methoxyethyl)-1-nonanol	202	70928-44-8	N	Not Detected	Not Detected	Detected
19.883	Propyl 2-methylbutyrate	144	37064-20-3	N	Not Detected	Not Detected	Detected
20.451	2,6,10-Trimethyltetradecane	240	14905-56-7	N	Not Detected	Not Detected	Detected
20.542	8-Hydroxylinalool	170	64142-78-5	N	Not Detected	Not Detected	Detected
20.591	(4-Hydroxy-3-methoxyphenyl)propane	166	2785-87-7	A	Not Detected	Not Detected	Detected
20.918	1-Tetradecanol	214	112-72-1	N	Not Detected	Not Detected	Detected
21.019	Tetradecane	198	629-59-4	N	Not Detected	Not Detected	Detected
21.091	2-(1-Methylene-2-propenyl)phenol	146	38865-47-3	A	Not Detected	Not Detected	Detected
21.708	3,4-Dimethoxyphenol	154	2033-89-8	A	Not Detected	Detected	Detected
21.895	4-Methoxy-3-(methoxymethyl)phenol	168	59907-65-2	A	Not Detected	Not Detected	Detected
22.237	1-Dodecanol	186	112-53-8	N	Not Detected	Not Detected	Detected
22.618	4-Acetyl-2-methoxyphenol	166	498-02-2	A	Not Detected	Not Detected	Detected
22.863	3,5-Di-t-butylphenol	206	1138-52-9	A	Not Detected	Detected	Not Detected
22.916	2,5-Di-tert-butylphenol	206	5875-45-6	A	Not Detected	Not Detected	Detected
23.249	4-(2,5-Dihydro-3-methoxyphenyl) butylamine	181	77515-67-4	B	Not Detected	Not Detected	Detected
23.316	4-Hydroxy-3-methoxyphenylethyl alcohol	168	2380-78-1	A	Not Detected	Not Detected	Detected
23.509	Vanillacetic Acid	182	306-08-1	A	Not Detected	Not Detected	Detected
23.639	Dodecanoic acid	200	143-07-7	A	Not Detected	Not Detected	Detected
24.015	Hexadecyl chloroacetate	318	52132-58-8	A	Not Detected	Detected	Not Detected
24.038	1-Hexadecanol	242	36653-82-4	N	Not Detected	Not Detected	Detected
24.087	Tridecane	184	629-50-5	N	Detected	Not Detected	Not Detected
24.091	Geranyl isovalerate	238	109-20-6	N	Not Detected	Detected	Not Detected
24.115	Hexadecane	226	544-76-3	A	Not Detected	Not Detected	Detected
24.261	3,4,5-Trimethoxyphenol	184	642-71-7	A	Detected	Not Detected	Not Detected

24.342	3'-Hydroxyquinalbarbitone	254	839-21-4	A	Not Detected	Detected	Not Detected
24.381	3,5-Dimethoxy-4-hydroxybenzyl alcohol	184	530-56-3	A	Not Detected	Not Detected	Detected
24.409	3,5-Dimethoxy-4-hydroxyphenethylamine	197	2413-00-5	A	Not Detected	Detected	Not Detected
24.481	3,4,5-Trimethoxyphenol	184	642-71-7	A	Not Detected	Detected	Detected
24.799	Vanillic acid	168	121-34-6	A	Not Detected	Detected	Not Detected
25.049	Ethyl vanillyl ether	182	13184-86-6	N	Not Detected	Not Detected	Detected
25.175	3,4-Dimethoxythiophenol	170	19689-66-8	A	Not Detected	Not Detected	Detected
25.199	Vanillyl ethyl ether	182	13184-86-6	A	Not Detected	Detected	Not Detected
25.459	Dodecylsuccinic acid anhydride	268	2561-85-5	A	Not Detected	Detected	Not Detected
25.459	Acrylic acid, dodecyl ester	240	2156-97-0	N	Not Detected	Not Detected	Detected
25.704	Benzoic acid, 2-hydroxyethyl ester	166	94-33-7	N	Not Detected	Not Detected	Detected
25.719	4-Allyl-2,6-dimethoxyphenol	194	6627-88-9	A	Not Detected	Detected	Not Detected
26.008	2-(Benzoyloxy)ethanol	166	94-33-7	A	Not Detected	Detected	Not Detected
26.306	4-Hydroxy-3-methoxycinnamic alcohol	180	458-35-5	A	Not Detected	Not Detected	Detected
26.393	Tetradecanoic acid	228	544-63-8	A	Not Detected	Not Detected	Detected
26.504	Oleic Acid	282	112-80-1	A	Not Detected	Detected	Not Detected
26.696	2-Isobutyl-5-isopentylthiophene	210	04/10/4806	A	Not Detected	Not Detected	Detected
26.769	1-Nonadecene	266	18435-45-5	N	Detected	Not Detected	Not Detected
26.793	3-Dodecyl-2,5-furandione	266	59426-46-9	A	Not Detected	Detected	Not Detected
26.831	1-Octadecanol	270	112-92-5	N	Not Detected	Not Detected	Detected
26.851	2,6-Dimethylheptadecane	268	54105-67-8	N	Detected	Not Detected	Not Detected
26.860	6-Methyloctadecane	268	10544-96-4	N	Not Detected	Detected	Not Detected
26.884	Nonadecane	268	629-92-5	N	Not Detected	Not Detected	Detected
26.947	3,5-Dimethoxy-4-hydroxyphenethylamine	197	2413-00-5	B	Not Detected	Detected	Detected
27.370	3-Methoxy-4-hydroxyphenylethyl amine	167	554-52-9	B	Not Detected	Not Detected	Detected
27.414	3,5-Dimethoxy-4-hydroxyacetophenone	196	2478-38-8	A	Not Detected	Not Detected	Detected
28.102	n-Nonenylsuccinic anhydride	224	28928-97-4	A	Not Detected	Not Detected	Detected
28.324	3,5-Dimethoxy-4-hydroxyphenylacetic acid	212	4385-56-2	A	Not Detected	Not Detected	Detected
28.415	1-Heptatriacotanol	536	105794-58-9	N	Not Detected	Not Detected	Detected
28.483	Hexadecanoic acid, methyl ester	270	112-39-0	N	Not Detected	Not Detected	Detected
28.541	2,3,4-Trimethoxybenzoic acid	212	573-11-5	A	Not Detected	Detected	Not Detected
28.569	7,9-Di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione	276	82304-66-3	N	Not Detected	Not Detected	Detected
28.907	n-Hexadecanoic acid	256	57-10-3	A	Detected	Not Detected	Not Detected
29.071	Ethyl 9-hexadecenoate	282	54546-22-4	N	Detected	Not Detected	Not Detected
29.104	Oleic Acid	282	112-80-1	A	Not Detected	Detected	Not Detected
29.167	Palmitic acid	256	57-10-3	A	Not Detected	Detected	Detected
29.307	Octadecanoic acid, ethyl ester	312	111-61-5	N	Detected	Not Detected	Not Detected
29.321	1-Nonadecanol	284	1454-84-8	N	Not Detected	Detected	Not Detected
29.352	Hexadecanoic acid, ethyl ester	284	628-97-7	N	Not Detected	Detected	Detected
29.365	6-Methyloctadecane	268	10544-96-4	N	Detected	Not Detected	Not Detected
29.388	2-Methyl-1-hexadecanol	256	2490-48-4	N	Not Detected	Detected	Not Detected
29.402	Nonadecane	268	629-92-5	N	Not Detected	Not Detected	Detected
29.581	2,5-Dimethoxybenzyl acetate	210	67698-75-3	N	Not Detected	Not Detected	Detected
29.927	Phenothrin	350	26002-80-2	N	Not Detected	Not Detected	Detected
30.231	Epoxyoleic acid	298	24560-98-3	A	Not Detected	Detected	Not Detected
30.361	2,4-Dimethoxybenzyl alcohol	168	7314-44-5	N	Not Detected	Not Detected	Detected
30.443	Hexadecanoic acid, propyl ester	298	2239-78-3	N	Detected	Not Detected	Not Detected
30.457	Isopropyl palmitate	298	142-91-6	N	Not Detected	Detected	Not Detected
30.707	3,5-Dimethoxy-4-hydroxyphenethylamine	197	2413-00-5	B	Not Detected	Detected	Not Detected
30.732	4-Hydroxy-3-methoxyphenylalanine	211	300-48-1	B	Not Detected	Not Detected	Detected
30.862	Methyl 5-(2 undecylcyclopropyl) pentanoate, trans	310	42199-20-2	B	Not Detected	Not Detected	Detected
30.862	Pentetic Acid	393	67-43-6	A	Not Detected	Detected	Not Detected
30.982	2-(2-Nitro-2-propenyl) cyclohexanone	183	78551-08-3	A	Not Detected	Detected	Not Detected
31.131	Linoleic acid	280	60-33-3	A	Not Detected	Detected	Detected
31.271	Linoleoyl chloride	298	7459-33-8	A	Not Detected	Detected	Not Detected
31.305	Ethyl-9,12-octadecadienoate	308	7619-08-01	N	Not Detected	Not Detected	Detected
31.314	Linoleic acid, ethyl ester	308	544-35-4	N	Not Detected	Detected	Not Detected
31.339	Oleic acid, ethyl ester	310	111-62-6	N	Detected	Not Detected	Not Detected
31.372	trans-13-Octadecenoic acid	282	693-71-0	A	Not Detected	Detected	Not Detected
31.426	Elaidic acid, ethyl ester	310	6114-18-7	N	Detected	Not Detected	Not Detected
31.43	Octadecanoic acid	284	57-11-4	A	Not Detected	Detected	Detected

31.479	17-Octadecynoic acid	280	34450-18-5	A	Detected	Not Detected	Not Detected
31.604	1-Docosene	308	1599-67-3	N	Detected	Not Detected	Not Detected
31.656	Octadecanoic acid, ethyl ester	312	111-61-5	N	Not Detected	Detected	Not Detected
31.661	Behenic alcohol	326	661-19-8	N	Not Detected	Not Detected	Detected
31.695	Heneicosane	296	629-94-7	N	Not Detected	Not Detected	Detected
32.142	Phorbol	364	17673-25-5	A	Not Detected	Detected	Not Detected
32.349	9-Hexadecenoic acid	254	2091-29-4	A	Not Detected	Detected	Not Detected
32.538	Oleic Acid	282	112-80-1	A	Detected	Not Detected	Not Detected
32.667	1-Heptatriacotanol	536	105794-58-9	N	Not Detected	Detected	Not Detected
33.226	Dihydroxanthin	308	31063-73-7	A	Not Detected	Detected	Not Detected
33.539	3-Hydroxydodecanoic acid	216	1883-13-2	A	Not Detected	Detected	Not Detected
33.718	2-Hexyldecanol	242	2425-77-6	N	Detected	Not Detected	Not Detected
33.731	2-Methyl-1-hexadecanol	256	2490-48-4	N	Not Detected	Detected	Not Detected
33.761	Lignoceric alcohol	354	506-51-4	N	Not Detected	Not Detected	Detected
33.765	Isovaleric acid, 3,7-dimethyl-2,6-octadienyl ester	238	109-20-6	A	Not Detected	Detected	Not Detected
34.107	Deoxysericealactone	276	19892-19-4	N	Not Detected	Detected	Not Detected
34.227	α-Santonin	246	481-06-1	N	Not Detected	Detected	Not Detected
34.531	1-Heptatriacotanol	536	105794-58-9	N	Not Detected	Detected	Not Detected
35.205	6-Hydroxy-buphanidrine	331	31128-91-3	A	Not Detected	Detected	Not Detected
35.678	Behenic alcohol	326	661-19-8	N	Detected	Not Detected	Not Detected
35.706	Octacosyl alcohol	410	557-61-9	N	Not Detected	Not Detected	Detected
36.139	4-Methoxy-2-methyl-1-nitrobenzene	167	5367-32-8	B	Not Detected	Not Detected	Detected
36.563	2,3-Dimethoxy-10,11-dihydrodibenzo[b,f]oxepin-10-ol	272	23396-52-3	A	Not Detected	Detected	Not Detected
36.682	17-Pentatriacontene	490	6971-40-0	N	Not Detected	Detected	Not Detected
37.646	Erucic acid	338	112-86-7	A	Not Detected	Detected	Not Detected
37.675	Octacosyl alcohol	410	557-61-9	N	Not Detected	Not Detected	Detected
37.877	Decanedioic acid, bis(2-ethylhexyl) ester	426	122-62-3	N	Not Detected	Detected	Not Detected
38.133	Squalene	410	111-02-4	N	Detected	Not Detected	Not Detected
38.629	Gibberellic acid	346	77-06-5	A	Not Detected	Detected	Not Detected
39.462	Methyl 13-hydroxykaur-16-en-18-oate	332	29444-14-2	N	Not Detected	Not Detected	Detected
39.568	3-Methoxy-4-hydroxyphenylethyl amine	167	554-52-9	B	Not Detected	Not Detected	Detected
39.784	17Alpha,21-dihydroxy-5alpha-pregnane-3,11,20-trione	362	1482-51-5	A	Not Detected	Detected	Not Detected
40.015	Ingol 12-acetate	408	51906-01-5	A	Not Detected	Detected	Not Detected
40.324	2-Methyl-1-hexadecanol	256	2490-48-4	N	Not Detected	Not Detected	Detected
42.23	1-Eicosanol	298	629-96-9	N	Not Detected	Not Detected	Detected
42.264	1-Hexacosene	364	18835-33-1	N	Not Detected	Detected	Not Detected
42.433	Ethyl iso-allocholate	436	NIST#: 43053	A	Detected	Not Detected	Not Detected
43.088	3,5-Dimethoxycinnamic acid	208	16909-11-8	A	Not Detected	Detected	Detected
44.219	17-Pentatriacontene	490	6971-40-0	N	Not Detected	Not Detected	Detected
45.37	Campesterol	400	474-62-4	N	Not Detected	Not Detected	Detected
46.251	Stigmasterol	412	83-48-7	N	Not Detected	Detected	Detected
45.462	Campesterol	400	474-62-4	A	Not Detected	Detected	Not Detected
47.356	α-Conidendrin	356	518-55-8	A	Not Detected	Detected	Not Detected
47.86	β-Sitosterol	414	83-46-5	A	Not Detected	Detected	Detected
48.062	Methyl gibberellate	360	510-50-9	N	Not Detected	Not Detected	Detected
48.351	Isobutylallylbarturic acid	224	77-26-9	A	Not Detected	Not Detected	Detected
48.895	Desoximetasone	376	382-67-2	A	Not Detected	Detected	Not Detected
50.017	2-Bromooctadecanal	346	56599-95-2	N	Not Detected	Not Detected	Detected

Table 3: Semi-Volatile Organic Compounds (BNAs) in various Spent Wash Sample

Most of the VOCs are found in spentwash samples and polluting the environment. These are isopropyl alcohol, butanol, ethyl acetate etc. Isopropyl alcohol is a colourless liquid, flammable chemical compound with a strong odour. It is used in making of cosmetics, pharmaceuticals, perfumes, dye solutions, antifreezes, soaps, cleaner and disinfecting agent. Prolonged exposure causes respiratory problems. On the other hand, between VOCs and nitrogen oxides (NOx) reactions occur in the presence of sunlight and result in photochemical oxidants (including ozone, peroxyacyl nitrates, peroxides, etc.) These chemicals can affect human health and are harmful to the environment, increasing harmfulness of NO to the environment by its oxidation to NO_2. Almost all VOCs directly contribute to global warming by absorbing infra-red radiation from sunlight and the more complex a VOC is, the greater its ability to absorb infrared radiation, yet most VOCs have a short atmospheric lifetime and are decomposed, thus diminishing their effect SVOCs are substances with a higher MW and boiling point than VOCs and it is less likely to become vapours at room temperature, but this does not mean they are any less hazardous to people and environment [52-55]. Benzyl alcohol is used as a solvent, a preservative, to manufacture chemicals, as a fragrance in perfumes and flavouring, and also as an ingredient in cosmetics. Benzyl alcohol is also used in inks, as a photographic developer and in dyeing nylon filament, textiles and sheet plastics. Exposure to very high concentrations could result in toxic effects such as respiratory failure, vasodilation, hypotension, convulsions, and paralysis.

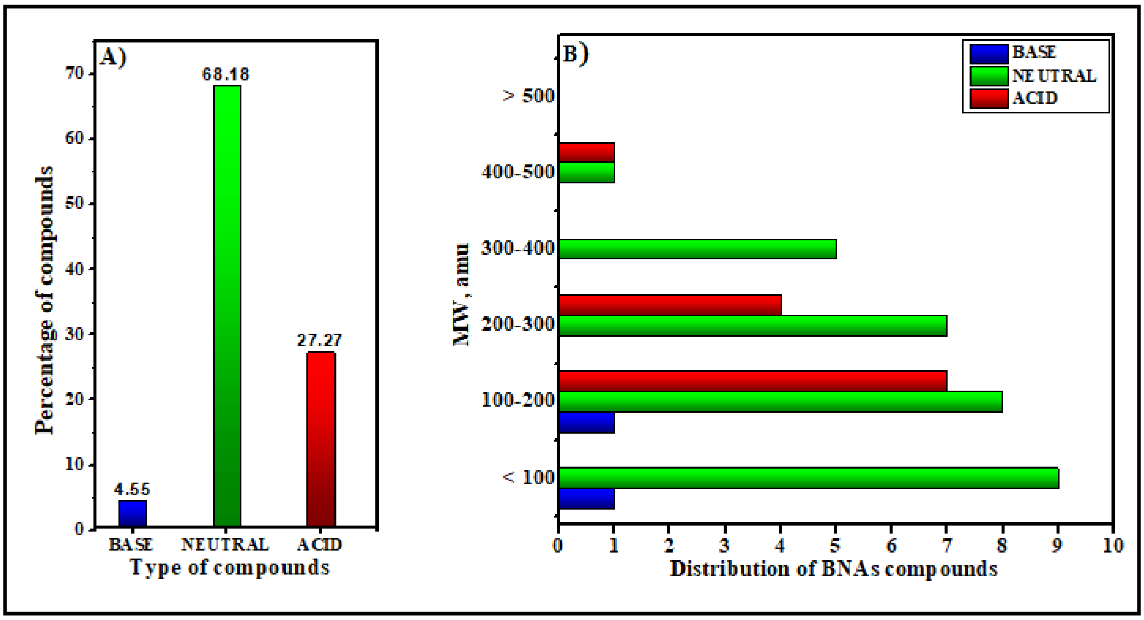

Figure 3: Percentage & Distribution of SVOCs in Juice Spentwash Sample (A) Percentage of BNAs Compound, B) Molecular Weight (MW) Distribution of SVOCs

The overall percentage distribution of forty four SVOCs in JSW sample is in the order like Neutral>Acid>Base. The Figure 3A shows that, neutral SVOCs like esters, alcohols, alkane and glycol compounds are conspicuously detected (68.18%) as compared to acidic (27.27%) and basic SVOCs (4.55%). Neutral SVOCs are un-ionized, hydrophobic and unaffected by low and high pH condition. In JSW sample (Figure 3B), the high molecular weight neutral SVOCs are not identified but noticeable detected in range of 100 to 400 amu. As the molecular weight increases, there is decline of neutral and basic SVOCs are observed. Basic SVOCs are detected in the range of 100-200 amu. The moderate & high molecular weight like acidic SVOCs are the second major detected compounds are in the range of 100-300 amu & 400-500 amu. Due to maximum neutral SVOCs, the pH of juice spentwash effluent sample is close to neutral.

Semi Volatile Compounds in Raw Spentwash (RSW) Sample

The TIC chromatogram of K-D extracted SVOCs sample demonstrate separation of analytes (Figure 2B). The chromatogram shows the one hundred & eleven compounds for 50 minutes data measurement which referring to few major components. Most of the 111 SVOCs were detected in RSW sample (Table 3) with qualitatively identification by using NIST library. The percentage wise distribution of base, neutral & acidic compounds are shown in Figure 4. Prominent acidic SVOCs are detected (53.45%) as compared to neutral (43.1%) and base SVOCs (3.45%).

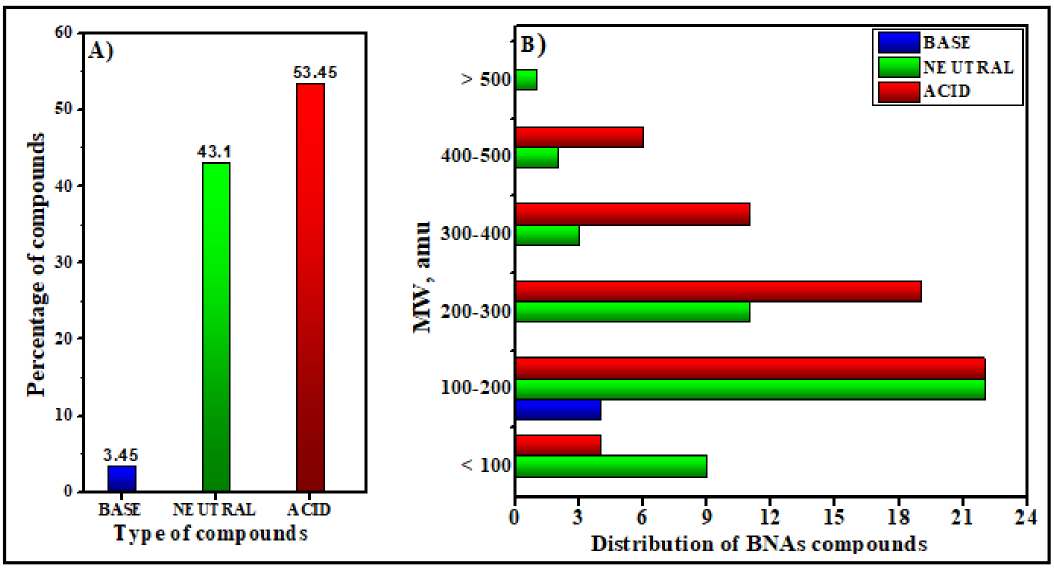

Figure 4: Percentage & Distribution of SVOCs of Raw Spentwash Sample (A) Percentage of BNAs Compound, (B) Molecular Weight Distribution of SVOCs

The acidic and neutral SVOCs are prominently detected in the range of 100-200 and 200-300 amu. The acidic SVOCs are detected in all MW ranges except >500 amu, whereas neutral SVOCs are detected in all MW range. Acidic SVOCs are extracted best under acidic pH because neutral compounds get protonated and more hydrophobic to partition into organic solvents. Liquid-liquid extraction at acidic pH recovers acidic and neutral SVOCs. Basic SVOCs are observed in the range of 100-200 amu. The presence of acidic SVOCs are prominent (53.46%) in all mw range will enhance the acidic pH of raw spentwash sample. Acidic and basic SVOCs are more water soluble and transportable because of their polarity and solubility, whereas neutral SVOCs are uncharged and unaffected by pH.

Untreated distillery wastewater changes pH level of the receiving water body of environment. Such changes can effect on the ecological aquatic system. The excessive acidity of waste water can result in release of hydrogen sulphide (H2S) to air. The compounds of low, medium & high MW are prominently observed under acidic and alcohol groups. The conspicuous compounds were detected are acidic in nature with MW from 154 to 362 are leads to acidic pH of sample. As molecular weight increases, the volatility decreases and compound is less likely to be gaseous at room temperature. Lower MW (<100 amu) compounds like solvents, alcohols & hydrocarbons are more volatile and easily transition into gas phase due to higher vapor pressure and more easily transported in the atmosphere. Most of the acidic and neutral compounds are produced during fermentation process and its concentration is depends on different process like pre and post treatments of cane milling, fermentation condition and type of distillation apparatus.

Semi Volatile Compounds in Biomethanated Spentwash (BSW) Sample

The TIC chromatogram of SVOCs in BSW sample (Figure 2C) determine good resolution for 50 minute data measurement. The SVOCs (140 compounds) with low, moderate & high molecular weight were detected in BSW sample (Table 3). Majority of 20-25 compounds peaks on chromatogram referring to major components like ester, alcohol and acids were characterised by high relative signal intensities.

The percentage wise distribution of base, neutral & acidic compounds are shown in Figure 5.

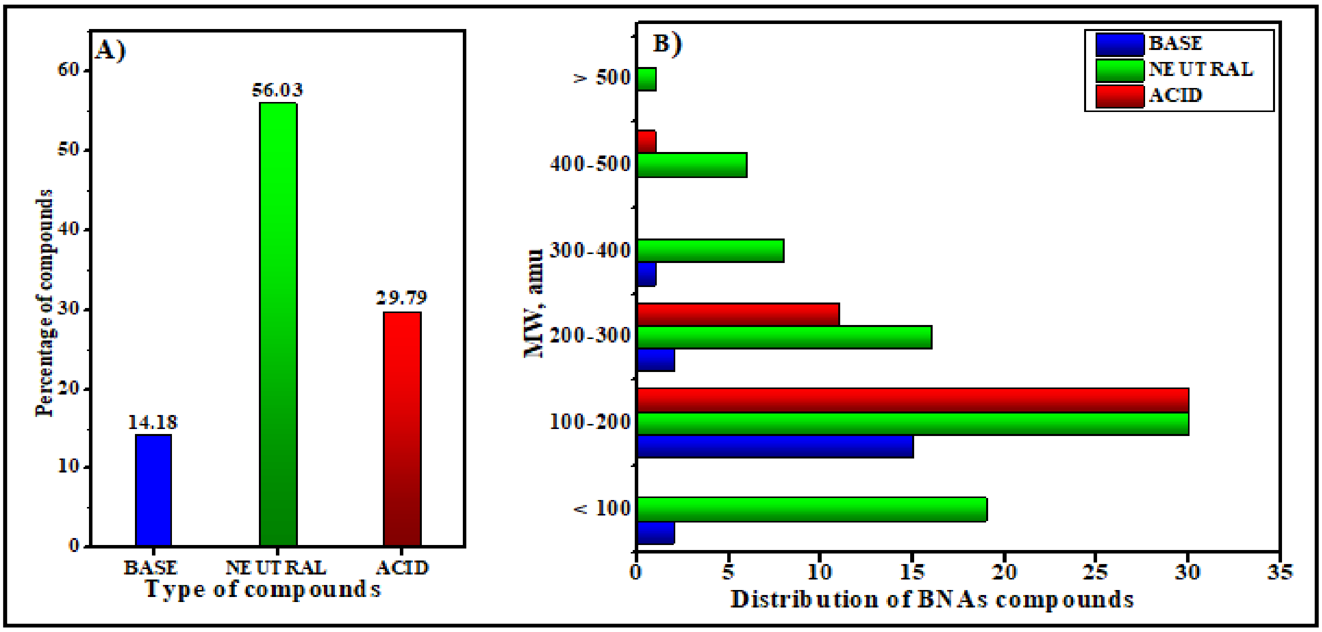

Figure 5: Percentage & Distribution of SVOCs of Biomethanated Spentwash Sample (A) Percentage of BNAs Compounds, (B) Molecular Weight Distribution of SVOCs

It is observed that, neutral SVOCs (56.03%) are prominently detected as compared to acidic (29.79%) and basic (14.18%) compounds in the range of 100-200 amu. The distribution of neutral SVOCs are detected in low, moderate and high mw range, whereas acidic SVOCs are prominently detected in range of 100-300 amu. The basic SVOCs are noticeable detected in mw range of 100-200 amu. The spreading of basic compounds at low mw range (<100 amu) & moderate mw range (200-400 amu) are observed. Basic SVOCs are extracted best under basic pH because mostly neutral compounds are unprotonated and more hydrophobic to get easier extract into organic solvents.

Conclusion

Anthropogenic pollutants contribute several environmental problems like air, soil & water pollution which causes great harm to ecosystem. The air pollution leading to respiratory illness, acid rain and climate change. The water pollution with low, moderate & high molecular weight compounds harming aquatic life and contaminating drinking water sources and soil pollution reducing fertility and making food chains toxic. The long term migration of SVOCs in gas & particle phases affects their environmental persistence, bioaccumulation and toxicological impact on ecosystem and human health. As MW increases, the vapor pressure decreases, making them less volatile and will favour in liquid phases. Untreated distillery wastewater with high TDS, suspended solids, high BOD & low DO, colour with acidic & alkaline pH wastewater would have adverse impact on ecosystem and make the water unsuitable for drinking, irrigation purpose. The Kuderna-Danish (K-D) is simple, economical and highly effective for concentrating extracts of SVOCs without losing the analytes in spentwash samples. The BNAs compounds in the 100-200 amu are prominently observed in juice, raw and biomethanated spentwash sample. In JSW & BSW sample, the neutral compounds are prominent than acidic compounds whereas, in RSW percentage of acidic compounds are prominent than neutral compounds. The JSW (44), RSW (111) & BSW (140) SVOCs (BNAs) are detected which leads to pH of spentwash sample. Alkaline nature of wastewater causes declination in plant and crop growth. In JSW & BSW waste water sample there is no harmful chemicals and has maximum percentage of neutral SVOCs, thus promoting soil health and plant growth. Acidic pH & recycling of raw spentwash waste water is corrosive and harmful to aquatic life and soil. The high molecular weight compounds are less volatile and thus normally have less odour. There is many medicinal compounds are detected in RSW & BSW samples. The SVOCs like phthalate, chlorophenyl, organochlorine pesticides, organo-phosphorous flame retardants (OPFRs), polychlorinated biphenyls (PCBs or PBBs) are not detected in distillery spentwash samples.

Moderate MW (100-500 amu) are less volatile than low MW compounds and can be transported over long distances in the atmosphere. High MW (>500 amu) compounds are low volatility at very high temperature and tend to settle and persist in soil or sediments for long periods in the presence of organic matter because they less volatile. Recycling of JSW and BSW water is possible due to maximum percentage of neutral compounds are detected as compared to RSW water. The formation of SVOCs in spentwash samples depends on different steps of the process like type of manual / mechanical harvest, the pre and post treatments of the cane during the milling, the alcoholic fermentation process, conditions and the type of distillation apparatus.

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