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Detection of Adulteration in Petrol Using Gas Chromatographic Technique

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ABSTRACT: Gasoline and Diesel are major transport fuels in India. Complaints on adulteration of these fuels at the point of sale or during transportation are a severe problem faced by the country. The present study on analysis of Gasoline by GLC and its comparison with the simulated samples of adulterated Gasoline reveals that the adulteration of Kerosene in Gasoline can be detected at a minimum level of 2%. In addition, it does not require any sample preparation. The method used has been found to be sensitive, specific to kerosene adulteration, rapid and economical for analysis of adulterated sample of petrol.

KEYWORDS: Gas Chromatograph, Adulteration, Petrol, Kerosene.

I.INTRODUCTION

The utilization of petroleum product in India is increasing day by day due to the growth of population urbanization and change in the life style. As the requirement increase the cost of the product also increase. Most of the times the expensive products are adulterated by blending with low quality cheaper hydrocarbons which have similar physical & chemical properties into high quality hydrocarbons .In case of petrol and diesel, mostly adulteration is done by adding kerosene & naphthalene(distillates of naphtha) because of their similar physical and chemical properties. It's not easy to distinguish between fuel and the adulterants. The common adulterants ,specifically for petrol are Naphtha, Benzene, Toluene, Pentane, hexane, SBP sprits, MTO, OCS 73, OCS 75, REMAX, SLOP OIL, C6 –C9 Raffinates, Resolve 77, Kerosene, Rexon, Pyrolysis Gasoline etc .Though fuel adulteration is an attempt to meet the price by adding cheaper solvents by beneficiary , air pollution is its ultimate result. Bharat stage emission standards are the emission standards instituted by the Govt. of India to regulate the air pollution from internal combustion of engine equipment, including motor vehicles. The standards and the timeline for implementation are set by the "Central Pollution Control Board" under the "Ministry of Environment & Forests and climate change". The standards are as follows:

Table 1: bs iv mg grade: mg91 is 2796:2014.

S.No.	Characteristics	Units	Requirement	
			Min.	Max.
1.	Colour, Visual			Orange
2.	Density at 15 ^{0}C	kg/m^3	720	775
3.	Distillation			
	a) Recovery up to 70^{0}C (E 70)	% by vol.	10	45
	b) Recovery up to 100^{0}C (E 100)	% by vol.	40	70
	c) Recovery up to 150^{0}C (E 150)	% by vol.	75	
	d) final boiling point (FBP)	^{0}C		210
	f)Recovery up to 70^{0}C (E 70)	% by vol.		2
4.	Research Octane Number (RON)		91	
5.	Motor Octane Number		81	
6.	Existent gum	g/m^3		40
7.	Sulphur , total	Mg/kg		50
8.	Lead content	g/l		0.005
9.	Benzene content	% by vol.		1
10.	Benzene content	% by vol.		1

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11.	Aromatic content	% by vol.		35
12.	Oxygen content	% by mass		2.7

In 2016, the Govt. of India has announced that the country would skip the BS V norms altogether and adopt BS VI norms by 2020.

In many laboratories adulteration in petrol is detected by checking the physical appearance or color and various characteristics like density, distillation (recovery at different temperatures), Research Octane Number, sulphur, lead, benzene, olefin, aromatic content etc.

In this paper, a chromatographic technique (Gas-liquid Chromatography) is used to study the effects of adding a known quantity of Kerosene to pure petrol and compare the results.

II. LITERATURE REVIEW

A method for detection of adulteration of biodiesel by soybean oil using UV-VIS spectrophotometer has been proposed [1].The observation of adulteration of diesel has been observed by using kinetic viscosity and opacity value as test parameters [2]. A field survey of excessive crankcase dilution of lubricating oil in petrol driven vehicle has done [3]. An approach to automatic fuel adulteration detection and reporting system has been proposed [4]. Fiber grating sensor technology has been used for detection of fuel adulteration [5, 6].

III. SAMPLING OF ADULTERATED PETROL

3.1. Guidelines

- Petroleum sample to be drawn preferably in cooler part of the day.
- Sampling equipment and sample containers must be rinsed properly with the product being sampled, till clear.
- The sample quantity collected shall be sufficient for carrying out the relevant tests and shall be filled up to v 85% capacity.

3.2. Safety Precautions

- Personnel should be made aware of potential hazards.
- All regulations covering entry into hazardous areas should be observed.
- Care should be taken to avoid breathing petroleum vapors during sampling operation.
- Proper disposal of product used for flushing

3.3. Sealing of sample

- Sample shall be properly sealed with tamper proof seal
- Details of seal (Seal No. and colour) or facsimile of seal shall be indicated on sample label as well as on forwarding letter. While applying sealing wax, naked flame shall be kept away from the petroleum vapors.
- DCP type fire extinguisher shall be kept at easy access
- Facsimile of the seal shall be indicated on sample label as well as on forwarding letter.

IV. INSTRUMENTAL METHOD

For this work, Chemito Instruments' GC 1000 with Iris 32 software is used. This instrument comprises of three basic parts, they are: injector, column and detector. In addition to these, three pressure gauges are there to control the flow of carrier gas, igniting gas and supporting gas. There is an oven to maintain the column temperature. The methodology applied for this work is as follows:

Table 2: Methodology for the Experiment.

1.Gases Required: I. Carrier gas N2 (343kpa to 392Kpa with more than 99.995% pure) II. Ignition gas H2 (294Kpa to 392Kpa with more than 98% pure) III. Supporting gas Zero Air (196Kpa to 392Kpa with Relative Humidity less than 85%)
2. Injection Temp. 2500C

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3. Oven Temp. Ramp 1- 40^{0}C Hold for 2 Min. increase @ 50C/Min Ramp 2- 70^{0}C Hold for 2 Min. increase @ 50C/Min Ramp 3- 150^{0}C Hold for 2 Min. increase @ 50C/Min Ramp 4- 200^{0}C Hold for 2 Min. increase @ 50C/Min Ramp 1- 250^{0}C Hold for 10 Min.
4.Column–OV 17 (Methyl 5% phenyl polysilozane) Length-8', Dia-(1/8)'' Max. Temp.-350^{0}C.
5.Detector Temp.300^{0}C

Figure 1: Gas Chromatograph (GC-1000).

V. EXPERIMENTAL ANALYSIS

For this work pure petrol and kerosene sample are taken. Then known quantities of kerosene are mixed with petrol and 10%, 5% and 2% adulterated petrol sample are prepared. Then they are injected into GC column and the following chromatograms are obtained. (Reproducibility of the instrument is very high. The graph was taken after running the same sample three times in GC. The result was same in each repetition.)

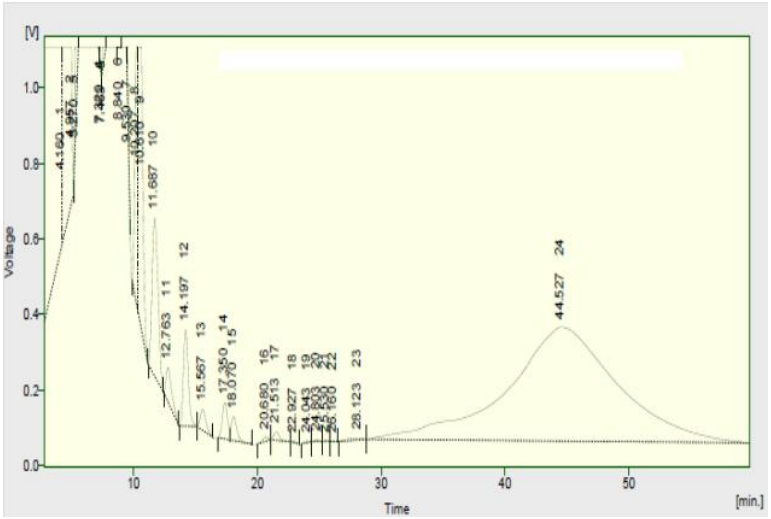

Figure 2: Chromatogram of pure petrol.

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Table 3: Result Table of Pure Petrol.

Sr.No.	Reten. Time [Min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [Min]
1.	4.160	181529.829	528.337	36.5	11.0	3.83
2.	4.957	21766.790	415.852	4.4	8.7	0.78
3.	5.270	2571.976	261.258	0.5	5.5	0.28
4.	7.320	351.564	56.235	0.1	1.2	0.17
5.	7.530	642.152	68.018	0.1	1.4	0.30
6.	8.840	6.022	0.701	1.210e-03	1.465e-02	0.15
7.	9.530	1580.816	198.224	0.3	4.1	0.16
8.	10.207	11804.803	676.717	2.4	14.1	0.29
9.	10.610	17678.550	746.653	3.6	15.6	0.37
10.	11.687	14166.934	418.202	2.8	8.7	0.55
11.	12.763	2959.386	91.207	0.6	1.9	0.44
12.	14.197	6991.566	254.419	1.4	5.3	0.43
13.	15.567	1795.502	55.405	0.4	1.2	0.48
14.	17.350	2748.294	96.353	0.6	2.0	0.46
15.	18.070	2083.961	63.233	0.4	1.3	0.57
16.	20.680	326.312	11.105	0.1	0.2	0.47
17.	21.513	735.092	22.375	0.1	0.5	0.49
18.	22.927	78.267	3.260	1.573e-02	0.1	0.41
19.	24.043	85.757	3.067	1.724e-02	0.1	0.47
20.	24.803	134.224	5.312	2.698e-02	0.1	0.42
21.	25.530	67.719	3.281	1.361e-02	0.1	0.35
22.	26.160	30.426	1.598	6.116e-03	3.341e-02	0.32
23.	28.123	549.347	6.184	0.1	0.1	1.11
24.	44.527	186068.547	301.444	37.4	6.3	8.42

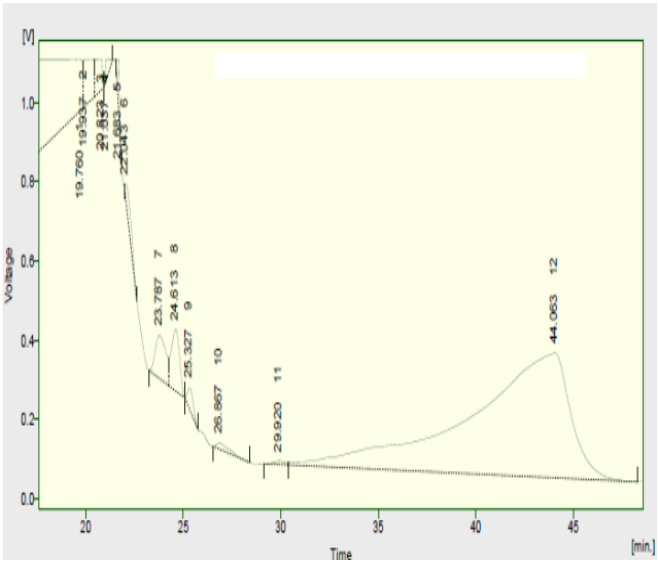

Figure 3: Chromatogram of Kerosene.

Table 4: Result Table of Kerosene.

	Reten.time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	19.760	687859.349	128.574	83.8	10.7	19.42
2	19.937	3899.471	119.980	0.5	10.0	0.61

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3	20.823	2033.580	76.781	0.2	6.4	0.41
4	21.037	671.067	55.749	0.1	4.6	0.37
5	21.683	1005.198	129.084	0.1	10.7	0.17
6	22.043	1558.265	35.017	0.2	2.9	0.19
7	23.787	4145.131	108.516	0.5	9.0	0.70
8	24.613	4633.805	155.010	0.6	12.9	0.53
9	25.327	1133.920	53.894	0.1	4.5	0.38
10	26.867	722.310	15.560	0.1	1.3	0.54
11	29.920	367.940	5.977	4.4858e-02	0.7	0.79
12	44.063	112346.835	314.917	13.7	26.2	5.02

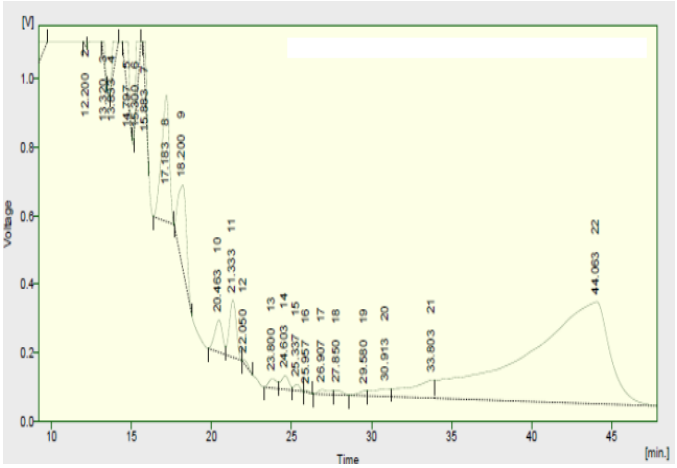

Figure 4: Chromatogram of 10% Adult Petrol.

Table 5: Result Table Of 10% Adulterated Petrol.

	Reten.time [min]	**Area [mV.s]**	**Height [mV]**	**Area [%]**	**Height [%]**	**W05 [min]**
1	9.507	296378.579	26.121	65.7	1.2	9.33
2	12.200	125.247	18.207	2.776e-02	0.9	0.21
3	13.320	630.556	73.590	0.1	3.5	0.20
4	13.833	1372.736	100.030	0.3	4.7	0.39
5	14.797	2993.669	175.322	0.7	8.2	0.44
6	15.300	2183.046	184.878	0.5	8.7	0.33
7	15.883	1523.981	165.246	0.3	7.8	0.19
8	17.183	12434.189	369.328	2.8	17.3	0.55
9	18.200	8571.801	242.919	1.9	11.3	0.76
10	20.463	3177.250	94.021	0.7	4.4	0.56
11	21.333	4994.795	167.511	1.1	7.9	0.48
12	22.050	280.070	9.943	0.1	0.5	0.26
13	23.800	964.180	26.604	0.2	1.2	0.68
14	24.603	1349.690	39.806	0.3	1.9	0.66
15	25.337	466.306	19.286	0.1	0.9	0.47
16	25.957	72.634	3.799	1.610e-02	0.2	0.31
17	26.907	749.737	13.322	0.2	0.6	1.01
18	27.850	461.281	12.560	0.1	0.6	0.60
19	29.580	492.115	14.038	0.1	0.7	0.56

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20	30.913	1781.482	23.460	0.4	1.1	1.53
21	33.803	5630.680	51.520	1.2	2.4	2.71
22	44.063.	104565.419	298.306	23.2	14.0	5.08

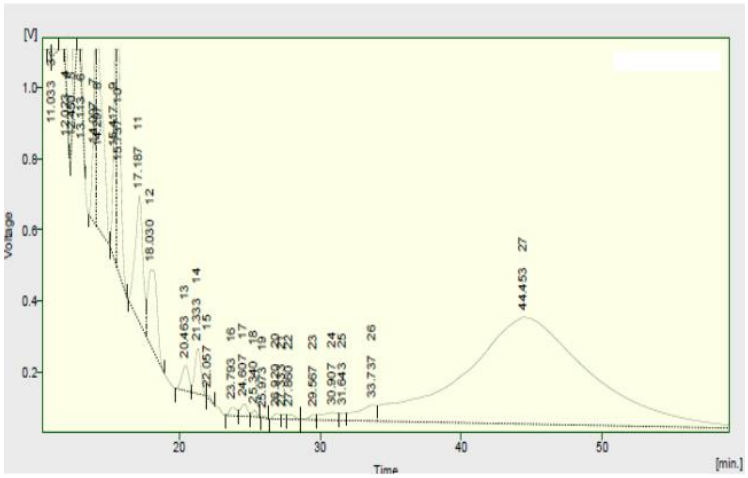

Figure 5: Chromatogram of 5% Adult petrol.

Table 6: Result Table Of 5% Adulterated Petrol.

	Reten.time [min]	**Area [mV.s]**	**Height [mV]**	**Area [%]**	**Height [%]**	**W05 [min]**
1	8.343	261391.860	27.545	48.6	0.7	8.15
2	10.837	134.408	18.328	2.499e-02	0.4	0.21
3	11.033	311.774	21.655	0.1	0.5	0.47
4	12.023	1354.833	152.413	0.3	3.7	0.20
5	12.450	2348.559	204.817	0.4	5.0	0.32
6	13.113	1454.797	155.839	0.3	3.8	0.20
7	14.007	7881.937	489.563	1.5	11.9	0.26
8	14.297	16381.829	506.813	3.0	12.3	0.55
9	15.417	9660.106	592.345	1.8	14.4	0.27
10	15.737	12430.908	630.924	2.3	15.4	0.30
11	17.187	13244.647	355.350	2.5	8.7	0.62
12	18.030	10034.273	217.233	1.9	5.3	0.79
13	20.463	2459.004	71.208	0.5	1.7	0.57
14	21.333	3815.735	127.709	0.7	3.1	0.48
15	22.057	203.518	8.147	3.784e-02	0.2	0.28
16	23.793	813.733	23.069	0.2	0.6	0.66
17	24.607	1173.960	34.576	0.2	0.8	0.60
18	25.340	423.987	17.504	0.1	0.4	0.43
19	25.973	88.689	4.734	1.649e-02	0.1	0.32
20	26.920	491.986	15.055	0.1	0.4	0.57
21	27.333	349.681	14.132	0.1	0.3	0.42
22	27.860	525.152	14.403	0.1	0.4	0.62
23	29.567	502.485	14.135	0.1	0.3	0.56
24	30.907	1692.168	21.592	0.3	0.5	1.55
25	31.643	689.771	21.387	0.1	0.5	0.55
26	33.737	4251.806	44.688	0.8	1.1	2.20
27	44.453	183599.086	299.241	34.1	7.3	8.47
28	95.210	49.650	0.009	9.233e-03	2.240e-04	0.01

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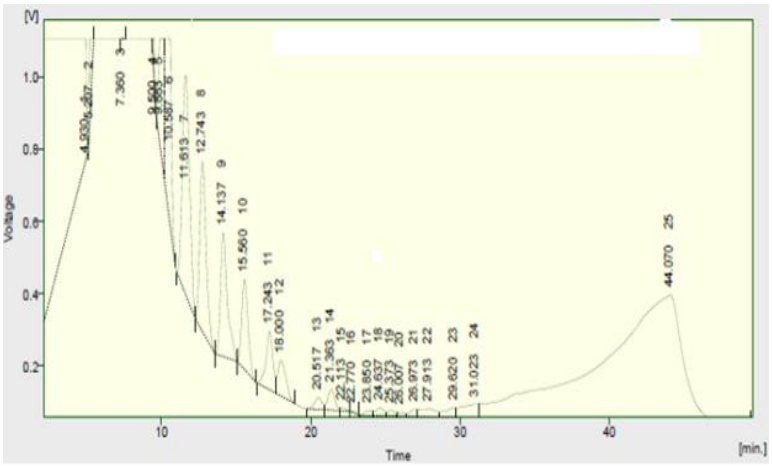

Figure 6: Chromatogram of 2% Adult petrol.

Table 7: Result Table Of 2% Adulterated Petrol.

	Reten.time [min]	**Area [mV.s]**	**Height [mV]**	**Area [%]**	**Height [%]**	**W05 [min]**
1	4.930	189947.645	336.381	44.7	8.3	4.58
2	5.207	2105.504	216.325	0.5	5.4	0.28
3	7.360	4.810	0.744	1.133e-03	1.844e-02	0.11
4	9.500	813.643	114.834	0.2	2.8	0.17
5	9.883	7750.990	290.961	1.8	7.2	0.43
6	10.587	14580.912	520.615	3.4	12.9	0.50
7	11.613	20527.997	608.437	4.8	15.1	0.54
8	12.743	14068.736	471.210	3.3	11.7	0.46
9	14.137	11070.684	343.849	2.6	8.5	0.45
10	15.560	7344.083	249.700	1.7	6.2	0.46
11	17.243	5243.963	161.858	1.2	4.0	0.48
12	18.000	3745.457	98.559	0.9	2.4	0.69
13	20.517	1189.839	33.647	0.3	0.8	0.58
14	21.363	1916.467	58.155	0.5	1.4	0.52
15	22.113	205.209	8.455	2.132e-02	0.2	0.41
16	22.770	90.516	3.935	2.132e-02	0.1	0.32
17	23.850	455.802	13.289	0.1	0.3	0.62
18	24.637	800.112	21.199	0.2	0.5	0.77
19	25.373	503.142	15.778	0.1	0.4	0.64
20	26.007	327.879	10.508	0.1	0.3	0.62
21	26.973	647.215	18.516	0.2	0.5	0.61
22	27.913	1649.824	21.865	0.4	0.5	1.45
23	29.620	1351.727	26.526	0.3	0.7	1.14
24	31.023	2918.442	37.308	0.7	0.9	1.50
25	44.070	135283.926	352.222	31.9	8.7	5.07

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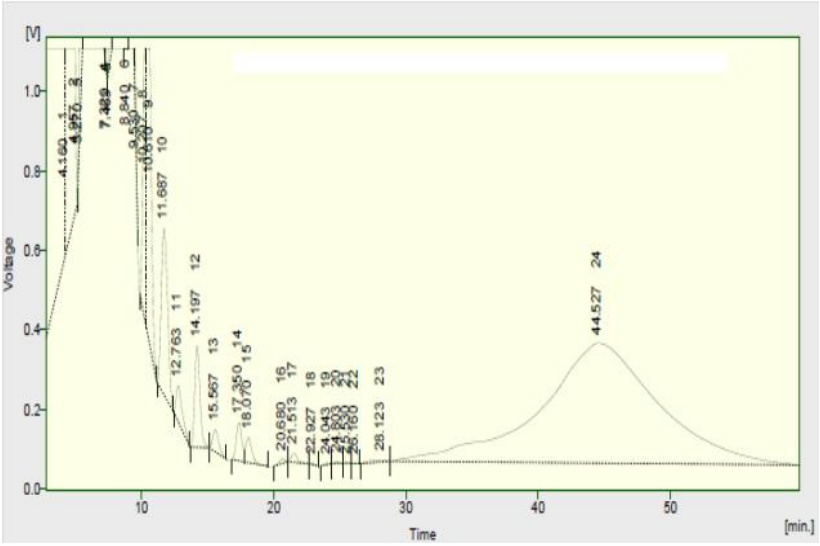

Figure 7: Chromatogram of 1% Adult petrol.

Table 8: Result Table Of 1% Adulterated Petrol.

	Reten.time [min]	**Area [mV.s]**	**Height [mV]**	**Area [%]**	**Height [%]**	**W05 [min]**
1	4.160	181529.829	528/.337	36.5	11.0	3.83
2	4.957	21766.790	415.852	4.4	8.7	0.78
3	5.270	2571.976	261.258	0.5	5.5	0.28
4	7.320	351.564	56.235	0.1	1.2	0.17
5	7.463	642.152	68.018	0.1	1.4	0.30
6	8.840	6.022	0.701	1.210e-03	1.465e-02	0.15
7	9.530	1580.816	198.224	0.3	4.1	0.16
8	10.207	11804.803	676.717	2.4	14.1	0.29
9	10.610	17678.550	746.653	3.6	15.6	0.37
10	11.687	14166.934	418.202	2.8	8.7	0.55
11	12.763	2959.386	91.207	0.6	1.9	0.44
12	14.197	6991.566	254.419	1.4	5.3	0.43
13	15.567	1795.502	55.405	0.4	1.2	0.48
14	17.350	2748.249	96.353	0.6	2.0	0.46
15	18.070	2083.961	63.233	0.4	1.3	0.57
16	20.680	326.312	11.105	0.1	0.2	0.47
17	21.513	735.092	22.375	0.1	0.5	0.49
18	22.927	78.267	3.260	10573e-02	0.1	0.41
19	24.043	85.757	3.067	10724e-02	0.1	0.47
20	24.803	134.224	5.312	2.698e-02	0.1	0.42
21	25.530	67.719	3.280	1.361e-02	0.1	0.35
22	26.160	30.426	1.598	6.116e-03	3.341e02	0.32
23	28.123	549.347	6.184	0.1	0.1	1.11
24	44.527	186068.547	301.444	37.4	6.3	8.42

After analyzing the graphs, it was found that the chromatogram of adulterated petrol sample is different than the pure one. Some peaks of kerosene like peaks with retention times 24.6, 25.3 and 26.6 etc. Also appears in case of adulterated sample. The comparison of 1% adulterated sample with the pure petrol is very difficult as most of the peaks are similar. Hence 2% adulteration can be considered as the minimum detection limit for this instrument.

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VI. CONCLUSION

The solvent adulteration detection is a very tough task as their compounds are also present in the original gasoline composition. Now-a-days, the detection of adulterated gasoline creates problem as some samples are approved but in reality they are adulterated. Hence with all the physico-chemical analysis currently used for the detection of adulteration, it is also recommended a cluster analysis. This practice will reduce the number of oil station gasoline samples approved when they are adulterated. After these analysis, if remain some doubt, it is recommended a more refined analysis, the gasoline gas chromatographic fingerprints of selected samples.

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REFERENCES

[1] DS David, AG Fernandes, et al. UV -Vis Spectrometric Detection of Biodiesel/Diesel Blend Adulterations with Soybean Oil, J. Braz. Chem. Soc., 2014; 25: 169-175.
[2] Sh R Yadav, VK. Murthy , et al. Estimation of petrol and diesel adulteration with kerosene and assessment of usefulness of selected automobile fuel quality test parameters, International Journal of Environmental Science &Technology, Dec 2005; 1: 253-255.
[3] Md Ehsan, Md Mahmudur Rahman, et al. Effect of fuel adulteration on engine crankcase dilution, Journal of Mechanical Engineering, December 2010; 41.
[4] V Jersha Felix, Purohitam Adithya et al. Fuel Adulteration Detection System, Indian Journal of Science and Technology, January 2015; 8: 90-95.
[5] V Mishra, SC Jain et al. Fuel adulteration detection using long period fiber grating sensor technique, Indian journal of pure and applied physics, Feb 2008; 46: 106-110.
[6] http://www.sciencedirect.com/science/article/pii/S0925400599001719