ECO-FRIENDLY DYEING OF COTTON FABRIC WITH A NATURAL DYE EXTRACTED FROM FLOWERS OF MUSSAENDA

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***Abstract*: The present investigation was carried out to revive the old art of dyeing with natural dye from flowers of *MUSSAENDA*. It belongs to family Rubiacea, commonly known as garden flower. The dye has good scope in the commercial dyeing of cotton in textile industry. In the present investigation, bleached cotton fabrics were dyed with different chemical mordents. Dyeing was carried out by pre-mordanting, post mordanting and simultaneous mordanting. Fastness properties of the dyed samples. The dyed samples have shown good washing, light and rubbing properties. The various color changes were measured by computer colour matching software. .Since they are eco-friendly naturally a available and giving no harm to the environment and the effluents created after the application on textiles for coloration gives less pollution.**
GC MS studies have proved that heavy metals such as antimony, arsenic, cadmium and lead were not present in the dye extract. Anti-bacterial and anti-fungal activities of the dye were also studied

***Keywords*: Extraction, Naturaldye, flowers, M*USSAENDA*, cotton ,textile**

INTRODUCTION

Natural dyes are generally eco-friendly and have many advantages over synthetic dyes. Natural dyes are known for their use in coloring of food substrate, leather, wood as well as natural fibers like cotton, silk, as major areas of application since ancient times. Natural dyes may have a wide range of shades and can be obtained from various parts of plants including leaves, seeds, roots, flowers, barks, fruit etc. Since the advent of widely available and cheaper synthetic dyes in 1856 having moderate to excellent colour fastness properties, the use of natural dyes having poor to moderate wash and light fastness has declined to a great extent. However, recently there has been revival of the growing interest on the application of natural dyes on natural fibers due to worldwide environmental consciousness [1].

The widely and commonly used synthetic dyes impart strong colour but causes carcinogenicity and inhibition of benthic photosynthesis [2].

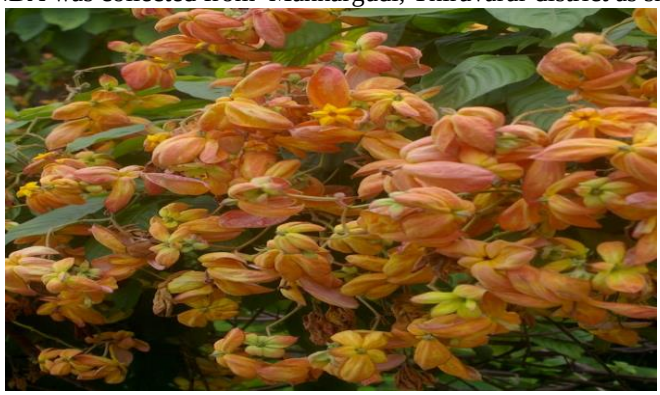
In many of the world's developing countries, natural dyes can offer not only rich and varied source of dye stuff, but also the possibility of an income through sustainable harvest and sale of the plants[3].

The present investigation was made on extraction and dyeing behavior of a natural dye obtained from the flower of the plant MUSSAENDA .by studying the various functions. such as dyeing time, dyeing temperature, MLR Ratio and dyeing method pre mordanating method is found to give better dyeing result during dyeing of cotton compared to simultaneous and post mordanating method. Amoung the different mordants employer $Feso_4$dyes,estimated at around$10x10^6$tons perineum, the production and application of which release vast amount of was tread unfixed colorants causing serious health hazards and disturbing the eco-balance of nature. Nowadays, fortunately there is increasing awareness among people towards natural dyes. Natural dyes are preferred in developed countries, because they are non-allergic, non-carcinogenic and have lower toxicity and better biodegradability than the synthetic dyes[5].

MUSSAENDA is a flowering plant, native to tropical regions of the America and Africa. It is a somewhat hairy shrub that when bruised gives a spicy pungent odour .The aromatic flowers are borne in clusters and are a mixture of red, yellow, white and orange florets. The leaves are pointed at the tip, rounded at the base and toothed in the margins. They grow as a bush andcanreachupto6feettalland wide. Deadheading spent flowers will encourage additional blooming and will prevent the growth of toxic berries. Lantanas are easy to care for and grow anywhere in well drained soils. Enjoy this attractive plant as butterflies enjoy the sweet nectar from the beautiful blooms. Flowers known to be haemostatic and the decoction of dried flowers are used for haemoptysis and pulmonary tuberculosis. Lantana leaves can be used for relief from headaches, fever, flu, coughs, colds toothaches and indigestion. It also relieves the symptoms of rheumatism and other joint pains. Use pounded fresh leaves applied as poultice for sprains, wounds and contusions. The dried lantana leaves that have been burned in a glass jar are also known to be a natural mosquito repellent [6].

MATERIALS AND METHODS

Source: The flowers of *MUSSAENDA* was collected from Mannargudi, Thiruvarur district as shown in figure



NAME - MUSSAENDA
FAMILY-RUBIACEA

Chemicals used: grade metallic salts such as copper sulphate, ferrous sulphate, alum, potassium dichromate, nickel sulphate and stannous chloride were used as chemical mordants.

Colour fastness:

The colour fastness of the dyed cotton fabrics were tested according to IS standards. Colour fastness to washing, light and rubbing were determined from standard test methods

RESULT AND DISCUSSION

Optimization of ethanolic extract of *MUSSAENDA*

Dye extraction: The flowers were soaked with 70% ethanol for 24 hours and then heated in a beaker kept over a water bath for 30 minutes to facilitate quick extraction. Then it was filtered and the filtrate was collected in a separate bottle.

Dyeing procedure: The cotton fabrics were dyed with dye extract keeping different M: L ratio such as 1:10, 1:20, 1:30 and 1:40. Dyeing was carried out different temperatures such as 40°C, 60°C and 80°C and continued for 1hour.

Mordanting: The cotton fabrics were treated with different chemical mordants by following three methods [7].

***(i) Pre-mordanting (PM)*:** In this method, cotton fabrics were pretreated with the solution of different chemical mordants and then dyed with dye extract.

***(ii) Postmordanting(POM)*:**In this method, dyed cotton fabrics were treated with solution of different chemical mordants.

***(iii) Simultaneous mordanting(SM)*:**In this method, the cotton fabrics were dyed with dye extract as well as different chemical mordants.

The flowers of *MUSSAENDA* were found to discharge colour in 70% ethanol very easily .Increasing the quantity of flowers 5g to 20g per100mLethanol and boiled for 30 minutes is accompanied with the increase in colour strength hand depth I colour[11].It was visually observed that colour of the dye extract was dark orange colour as shown in figure3.

Fig.3: Ethanolic extract from flowers of Mussaenda

Effect of mordanting:

The dye extract was found to be suitable for cotton fabric. The cotton fabrics were dyed with different chemical mordants. It was observed that the dye uptake was found to be good in pre mordanting (PM) method is shown in figure4.

Fig.4: Surface color strength (K/S values) of dyed cotton fabrics after pre (PM), post (POM) and simultaneous mordanting (SM)

Effect of M: L ratio:

The cotton fabrics were dyed with dye extract keeping various M:L ratio such as 1:10,1:20,1:30 and 1:40.It was observed that, the dye uptake was good in M: L ratio1:30.

Mussaenda dye extracted for 30 minutes, 1hour, 2hour using temperature after 60^0c, 80^0c, 90^0c, The optimum temperature was selected for extraction. From the optical density value.

Three method of mordanting, Pre, Post, Simultaneous used with each mordant Six metal mordant $CuSO_4$, $AlSO_4$, $FeSO_4$, $K_2Cr_2O_7$, $NiSO_4$,$ZnSO_4$ and myrobalan selected for mordanting for mordanting cotton fabric during dyeing

PRE MORDANTING:

- Treatment with metal salts:

The wetted out cotton fabric were treated with metal salt using the following recipe.

Recipe:

Metal Salt	-	3% ($FeSo_4$)
Myrobalan	-	(4:1) Ratio
MLR	-	1:30
Temperature	-	60^0c
Time	-	1 hour

Procedure:

Accurately weighted cotton samples were entered into different both containing required amount of metal salt and water. The treatment was carried out for 1 hour at 60^0c. The samples were taken out squeezed and using dying procedure without washing.

POST MORDANTING METHOD:

- Dyeing with dye extracts

The wetted out cotton samples were dyed with dye extracts following recipe.

Recipe:

Dye Extract	-	3%
MLR	-	1:30
Temperature	-	60^0C
Time	-	1hour

Procedure:

The wetted cotton sample were entered into different baths containing required amount of dye and water .The dyeing was carried out for 1hour at 60^0C. The dyeing samples were taken out raised with water squeezed and dried.

Treatment with metal salts:

The dyed cotton sample were treated with metal salt($FeSO_4$) using following recipe

Recipe:

Metal Salt	-	3%($Feso_4$)
Mordants	-	4:1(Ratio)
MLR	-	1:30
Temperature	-	60^0C
Time	-	1hour

Procedure:

After treating with metal salts the sample was taken out were rinsed with water squeezed

Simultaneous Mordanting:

The wetted out cotton sample were treated with dye extracts and metal salt simultaneously using the following recipe

Recipe:

Dye Extract	-	3%
Metal Salt	-	3%(FeSO4)
Mordants	-	4:1(Ratio)
MLR	-	1:30
Temperature	-	60^0c
Time	-	1hour

Procedure:

The wetted cotton samples were entered into different baths containing required amount of dye, mordant mixture $FeSO_4$ and myrobalan 4:1 and water. The dyeing was carried out for 1hour at 60^0C. The dyeing samples were taken out rinsed with squeezed and dried.

The Same procedure was followed for metal mordant $Feso_4$ and Myrobalan ratio 1:4 to Pre ,Post, and Simultaneous method.

The dyeing of cotton using mordant $Feso_4$ and Myrobalan procedure were followed the remain in mordant's $Cuso_4$,$Znso_4$, $K_2Cr_2O_7$, $NiSO_4$,and $AlSO_4$…

Characterization of Natural Dye:

The Ethonolic extract of dye obtained from the plant mussaenda the following GC-MS instrument for analysis

INSTRUMENT DETAILS:

MAKE	: PerkinElmer Clarus 500
SOFTWARE	: Turbomass ver5.2.0
Capillary Column Elite-5MS(5% Phenyl 95% dimethylpolysiloxane)	
Column length	: 30m
Column id	:250Mm
GC Condition Oven Program	:50^0C(2mins)@8^0C/min to 200^0C(3min)@8^0C/min to270^0C(10min)

Injector temp	: 280^{0}C
Carrier gas	: Helium @flow rate 1ml/min
Split	:1: 10
MSConditions	
Mass range	:40-600amu
Type of ionization	: Electron Ionisation(EI)
Electron energy	:70ev
Transfer line and source	
Temperature	:200^{0}C,180^{0}C
Library	:NIST 2005
Sample injected	:2.0microlitres

Fastness properties:

The fastness properties of dyed cotton fabrics were evaluated by standard IS methods. It was observed that, dyeing with *Lantanacamara Linn* gave good fastness properties. The fastness properties of dyed cotton fabrics are shown in table 3. Overall, it could be used for commercial purposes and attain acceptable range.

PROCEDURE FOR LIGHT AND WASHING FASTNESS:

LIGHT FASTNESS:

Procedure:

Light fastness was done by the dyed materials was exposed to sunlight.
A cardboard sheet has to be taken and covered with black paper. The dyed materials were cut and fix the all samples on the black card board.

Half of the samples were covered with a brown paper. This setup was kept in bright sunlight for 1 hour.

The brown paper was removed after 24 hours and compared the exposed and unexposed part of the dyed materials. Comparison should be made with the following.

Grade 1	-	very poor
Grade 2	-	poor
Grade 3	-	fair
Grade 4	-	good
Grade 5	-	Excellent

Washing Fastness:

Recipe:

Soap solution	-	5%
Time	-	1 hour

Procedure:

The dyed fabric piece is sandwiched between two un dyed piece and stretched along the edges. This composite piece is placed in a soap solution and treated for 1 hour.

The washing fastness grade are given after absorbing the loss of color from dyed piece and staining of the adjacent un dyed piece

Grade 1	-	very poor
Grade 2	-	poor
Grade 3	-	fair
Grade 4	-	good
Grade 5	-	Excellent

Table 3: Fastness properties for cotton fabric

METHODS	METAL SALT	RATIO	WASHING FASTNESS	LIGHT FASTNESS	WASHING FASTNESS	LIGHT FASTNESS
PRE MORDANTING	$CuSO_4$	4:1	5	4		
		1:4	3	2		
	$FeSO_4$	4:1	5	5		
		1:4	4	3		
	$ZnSO_4$	4:1	5	4		
		1:4	3	2		
	$K_2Cr_2O_7$	4:1	5	5		
		1:4	2	3		
	$NiSO_4$	4:1	4	5		
		1:4	2	2		
	$AlSO_4$	4:1	5	5		
		1:4	3	3		

METHODS	METAL SALT	RATIO	WASHING FASTNESS	LIGHT FASTNESS	WASHING FASTNESS	LIGHT FASTNESS
POST MORDANTING	$CuSO_4$	4:1	5	4		
		1:4	2	2		
	$FeSO_4$	4:1	5	4		
		1:4	3	3		
	$ZnSO_4$	4:1	5	5		
		1:4	3	3		
	$K_2Cr_2O_7$	4:1	5	5		
		1:4	3	2		
	$NiSO_4$	4:1	5	4		
		1:4	3	2		
	$AlSO_4$	4:1	5	5		
		1:4	3	3		

METHODS	METAL SALT	RATIO	WASHING FASTNESS	LIGHT FASTNESS	WASHING FASTNESS	LIGHT FASTNESS
SIMULTANEOUS MORDANTING	$CuSO_4$	4:1	5	4		
		1:4	2	2		
	$FeSO_4$	4:1	4	5		
		1:4	2	3		
	$ZnSO_4$	4:1	5	5		
		1:4	3	3		
	$K_2Cr_2O_7$	4:1	5	5		
		1:4	2	2		
	$NiSO_4$	4:1	4	5		
		1:4	3	2		
	$AlSO_4$	4:1	5	4		
		1:4	3	3		

GCMS studies:

The presence of heavy metals like antimony, arsenic, cadmium and lead in the dye extract causes dermatological problems to

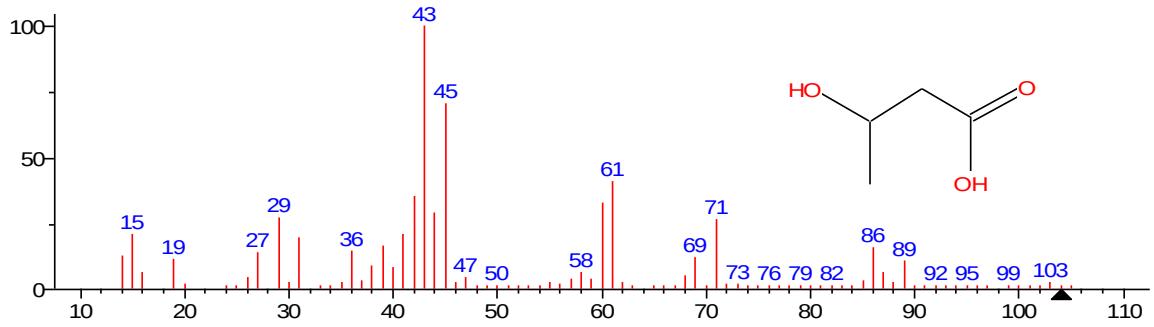
the wearer and also eco- friendly dye should not contain these heavy metals [10]. The presence / absence of these heavy metals were tested by GCMS (Gas chromatography mass spectrometry)

The GC- MS study indicate the following fragments were present in the natural dye table

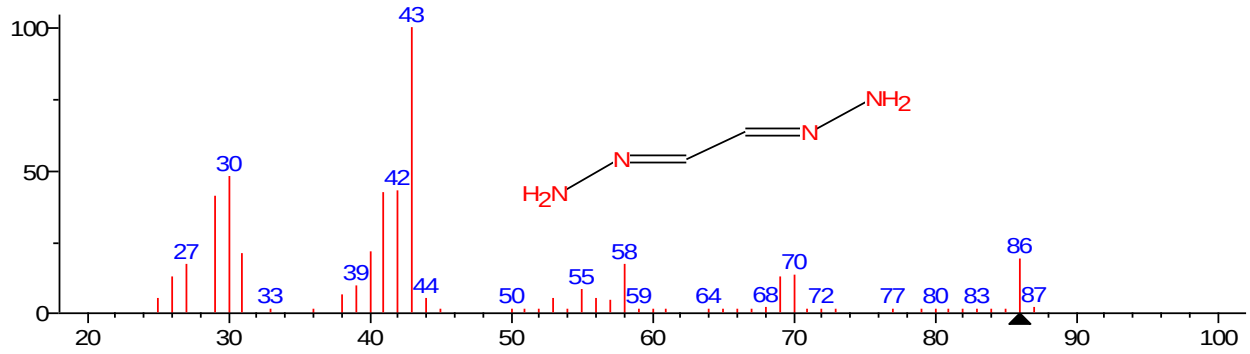
List of fragmented compounds for the sample

S.No.	Peak Name	Reaction Time	Peak area	% peak area
1.	Name: Butanic acid, 3-hydroxy- Formula: $C_4H_8O_3$ MW : 104	2.55	4405816	4.4780
2.	Name:1-piperidinecarbonitrile Formula:$C_6H_{10}N_2$ MW: 110	8.05	297834	0.3027
3.	Name: Ethane-1,2-dimine,N,N'-diamino Formula: $C_2H_6N_4$ MW: 86	8.69	034498	0.3095
4.	Name: phenol Formula:C_6H_6O MW:94	9.05	1704477	1.7324
5.	Name:5H-1,4-Dioxepin,2,3-dihydro-2,5- Dimethyl Formula: C7H12O2 MW:128	9.69	7916059	8.0457
6.	Name:2,5-Dimethyl-4-hydroxy-3(2H) Furanone Formula:C6H8O3 MW:128	10.49	3388234	10.49

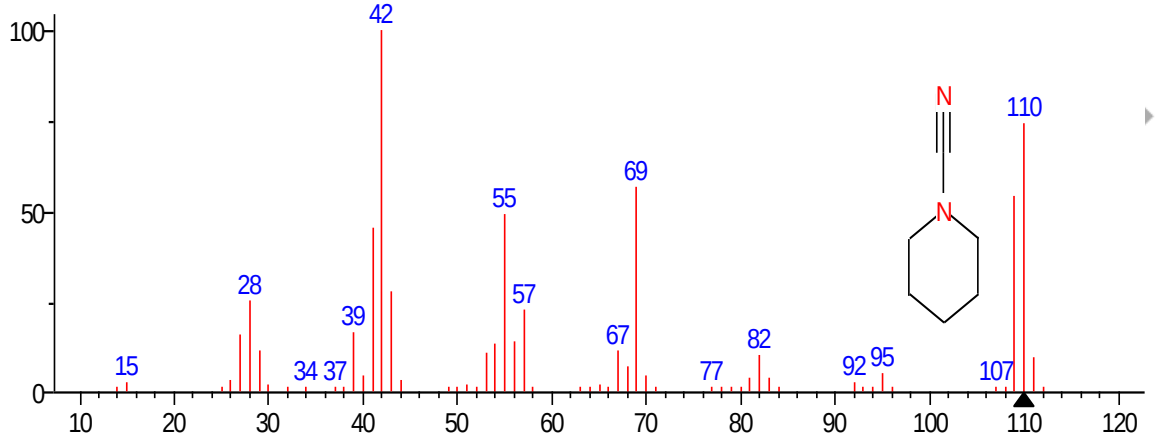
7.	Name:4-Hexenoic acid, 2-amino-6- Hydroxyl-4-metyl Formula: C7H13O3 MW:159	11.13	1155160	1.1741
8.	Name:a'-L-Galactopyranoside, methyl 6-deoxy- Formula: C7H14O5 MW:178	11.48	3149456	3.2011
9.	Name: 4H-pyran-4-one,2,3- Dihydro -3,5-dihydroxy -6- Methyl- Formula: C6H8O4 MW:144	11.92	787527	0.8004
10.	Name:Benzoic acid, 2-hydroxy-,methyl Ester Formula: C8H8O3 MW:152	12.55	788782	0.8004



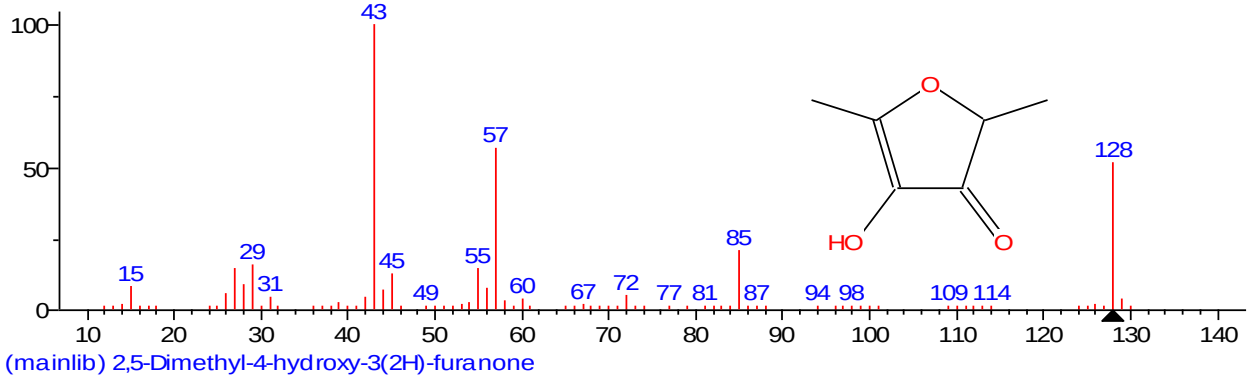
(mainlib) Butanoic acid, 3-hydroxy-



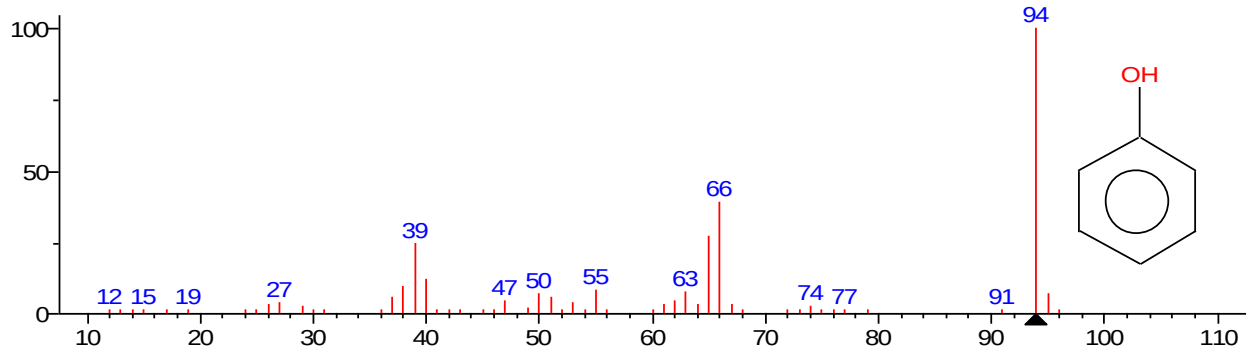
(mainlib) Ethane-1,2-diimine, N,N'-diamino-



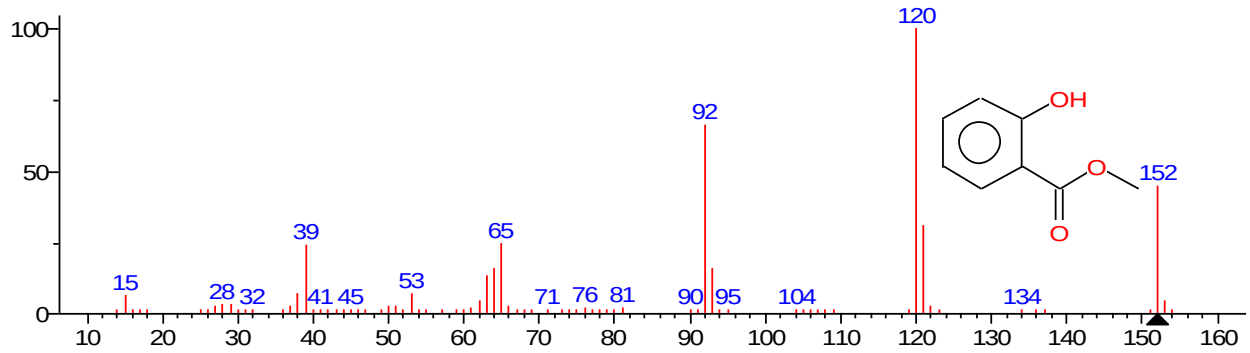
(mainlib) 1-Piperidinecarbonitrile



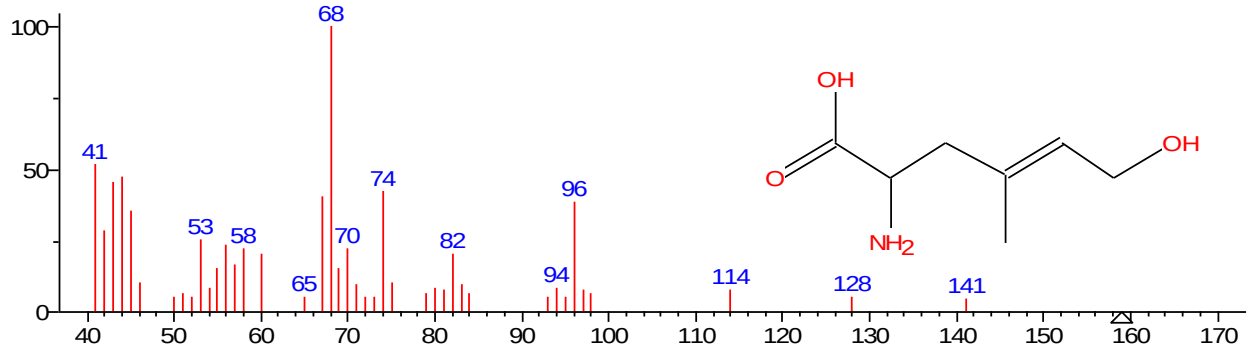
(mainlib) 2,5-Dimethyl-4-hydroxy-3(2H)-furanone



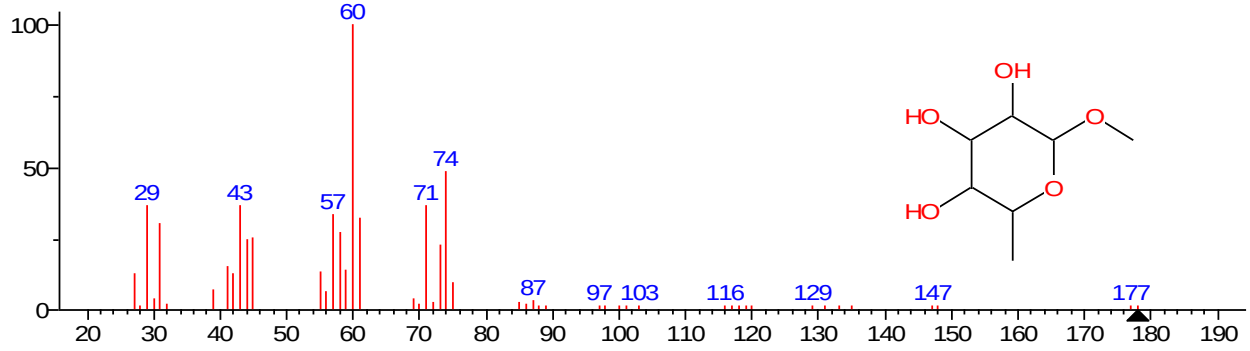
(mainlib) Phenol



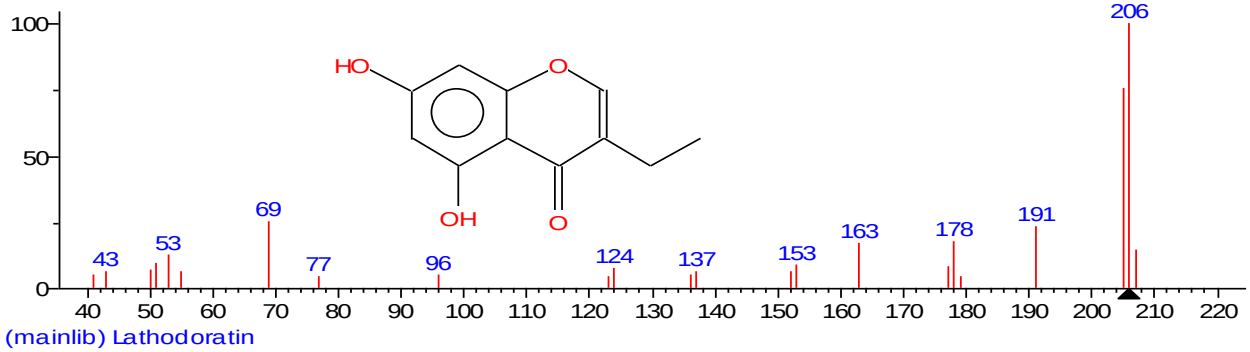
(mainlib) Benzoic acid, 2-hydroxy-, methyl ester



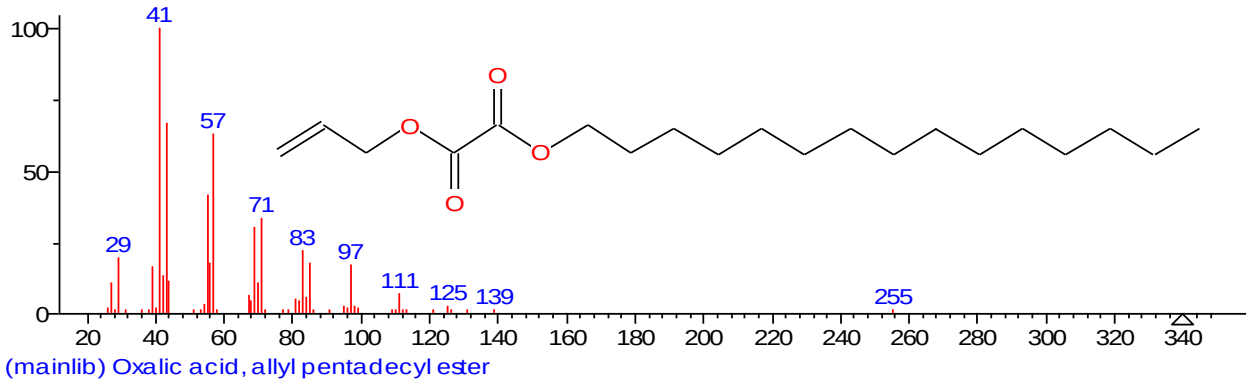
(mainlib) 4-Hexenoic acid, 2-amino-6-hydroxy-4-methyl-



(mainlib) à-L-Galactopyranoside, methyl 6-deoxy-



(mainlib) Lathodoratin



CONCLUSION

In the present study and attempt dyeing variable for dyeing of cotton with natural dye extract from MUSSANDA and also test the fastness of dyed samples. Based on the percentage of absorption.

a. Alkaline medium on (EtOH+Na2CO3)
b. 60 minutes extraction time and
c. Temperature 80^0C

Were optimized condition for extraction of natural dye
From the OD value and percentage absorption it was concluded the dyeing of Mussaenda using MLR 4:1 (Myrobalan and metal salt) gave bright shade when compared to MLR 1:4

- The Fastness study also reveals that dyeing using MLR 4:1 was the suitable condition for better shade
- The GC-MS result reviews that the presents of hydroxyl groups present in the structure of the natural dyes

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