A novel wool dye synthesised via recycling of side fraction of petrol: metal naphtenates (cyclohexane dicarboxylates)

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ABSTRACT – REZUMAT

A novel wool dye synthesised via recycling of side fraction of petrol: metal naphtenates (cyclohexane dicarboxylates)

The study aims to produce naphthenates (cyclohexane dicarboxylate) obtained from side fractions of petrol and used as dyestuff in the textile sector to abate waste-oriented pollution and to reutilize petroleum-based wastes. The investigations showed that obtaining blue dyestuff from copper, red dyestuff from cobalt and yellow dyestuff from nickel was possible. Moreover, it was determined that wool fabric could be dyed with good colour fastness with the dyestuffs mentioned. Therefore, this will help to reduce environmental pollution by addressing a novel and beneficial area of usage for naphthenates, produced via waste recycling.

Keywords: *petrol, naphthenate, dye, wool, recycling*

Un nou colorant pentru lână sintetizat prin reciclarea fracției secundare a benzinei: naftenați metalici (ciclohexan dicarboxilaţi)

Studiul își propune să producă naftenați (ciclohexan dicarboxilat) obținuți din fracții secundare ale benzinei și să fie utilizați sub formă de coloranţi în sectorul textil pentru a reduce poluarea orientată spre deșeuri și pentru a reutiliza deșeurile pe bază de petrol. Investigațiile au arătat că este posibilă obținerea de colorant albastru din cupru, colorant roșu din cobalt și colorant galben din nichel. Mai mult, s-a stabilit că materialul textil din lână poate fi vopsit pentru a avea o bună rezistență a culorii cu coloranții menționați. Prin urmare, acest lucru va ajuta la reducerea poluării mediului prin abordarea unui domeniu nou și benefic de utilizare a naftenaților, produși prin reciclarea deșeurilor.

Cuvinte-cheie: *benzină, naftenat, colorant, lână, reciclare*

INTRODUCTION

The Textile Industry is among the industries that cause the most environmental pollution. Dyeing methods used for the dyeing process in the textile sector and dyestuff variety are increasing with the changing and renewing technology. When the reasons for the increase in dyestuff variety are investigated, economy and environmental awareness may come first with the development of modern technology and industrialisation, there is an increase in the amount of waste that can pose a threat to living and environmental health. Increasing the cancer ratio each year is proof that wastes hurt human health. Recycling of wastes has gained great importance by utilising petroleum derivatives and abietic acid residuals known as wastes. This is significant for both reusability of wastes and preventing environmental pollution [1–3].

Many of the chemicals used in our daily lives are unsustainably derived from petroleum [4]. Petrol, one of the most precious subsurface raw materials in the world, provides benefits to humankind not only in technology but also in several other areas. Utilization areas of materials produced as a side fraction of petrol have been becoming increasingly crucial. Some side fractions do not have great value whereas others could be evaluated and sold after an additional process [5].

Dicarboxylic acids found in petroleum wastes are acid groups that contain two carboxyl groups and have the general formula $CnH2n(CO_2H)_2$. Their common feature is being acidic, those that dissolve in water give an acidic reaction. The relative position of the carboxyl group affects the physical and chemical properties of the molecule. Dicarboxylic acids show the familiar chemical behaviour of monocarboxylic acids. Dicarboxylic acids are solids and are more soluble in water than monocarboxylic acids with the same number of carbons. The lower members of the row dissolve in water and are less soluble in organic solvents, and the solubility decreases as the row progresses. In monocarboxylic acids, a polymeric structure is formed by combining the carboxyl groups at the two ends with hydrogen bonds in dicarboxylic acids, as in the formation of dimer molecules by the bonding of carboxyl groups with hydrogen bonds [6].

Naphthenates are derived from the side fractions of petrol, which are normally in the waste class. In recent years, recycling gained high importance to decrease waste-related pollution. In our previous study, we examined the application of silver naphthenates (silver monocarboxylate, silver dicarboxylate and silver abietate) on cotton fabric surfaces, their antibacterial effects, and their washing resistance. Silver naphthenate was applied to cotton surfaces by impregnation method. Attitudes towards selected gram-negative and gram-positive bacteria were examined after 5, 10, 15 and 20 washes**.** [7]

This study, it was aimed to investigate the possibilities of using cyclohexane dicarboxylates, which are included in the metal naphthenates class and obtained as waste from the side fraction of petroleum, as a dyestuff in the textile industry. In our previous study, copper naphthenate dye was synthesized from the aforementioned waste and it was determined that this dye is suitable for dyeing wool [8]. As it is known, dyes for three main colours (yellow, red, and blue) are necessary to carry out trichromy dyeing in the textile field. For this reason; the current study, it was aimed to obtain dyes from metal complexes of cyclohexane dicarboxylates (copper, cobalt and nickel) giving yellow, red and blue colours. Therefore, a new application field, which will help to prevent environmental pollution, was introduced by developing a beneficial usage for waste, naphthenates, after recycling.

MATERIAL AND METHOD

Copper, cobalt and nickel cyclohexane dicarboxylate complexes were obtained from the reaction of naphthenic acid obtained from the side fraction of petrol and metal (Me) salts of copper, cobalt and nickel. The synthesis reaction of the metal cyclohexane di carboxylate compound is as follows:

$$2RCOOH + 2NaOH + MeCl_2 \rightarrow Me(RCOO)_2 + \\ + 2NaCl + 2H_2O$$

For actualization of the reaction, firstly cyclohexane dicarboxylic acid was dissolved in an organic solvent and acids sodium salts were constituted by adding caustic soda [6–7]. Then copper, cobalt and nickel cyclohexane di carboxylates were obtained by adding copper chloride, cobalt chloride and nickel chloride respectively [8].

A thermometer, condenser and dropping funnel were placed at the necks of a three-necked flask. Then, the solution of the cyclo hexane dicarboxylic acid (Sigma Aldrich) in 10% (v) diethyl ether, which was calculated stoichiometrically, was put into the flask. The solution of 10 % (v) NaOH was put into the dropping funnel. The temperature was raised to 40–45°C and the solution was stirred while dropping NaOH from the dropping funnel for 60 minutes. The pH of the medium was adjusted between 7 and 8. Then the heater was turned on, the dropping funnel was opened and the mixture was stirred by running an electromagnetic stirrer (Hot-Plate 300°C 15 cm circular M15 type) at room temperature for 1 hour, then the solution was kept for 24 hours. The solution obtained was put into the extraction flask and the liquid phase was separated from the organic phase. After the removal of solvent from the organic phase, a metal cyclohexane di carboxylates compound was obtained [9–13]

After the synthesis of cyclohexane di carboxylates dyes, optimum dissolution conditions for synthesized dyes were determined and their usability in wool dyeing was investigated.

Determination of the optimum dissolution conditions for synthesized dyes

To use a dye for the colouring of textile fibres, a solution or a dispersion of the dye should be prepared. Furthermore, in the case of the water solubility of the dye, to determine the maximum intensity of the dye that can be carried out, the upper limit of solubility (maximum solubility) needs to be known. Thus, to determine the optimum dissolution conditions for the synthesized dyes in this study, solutions:

- with 5 different dispersing agent concentrations (0.5; 1; 1.5; 2.0; 2.5 g/l)
- at 5 different pH values (1; 4; 7; 10; 13) and
- with 5 different dye concentrations (2; 4; 6; 8; 10 g/l)

were prepared. As it was observed in our washing fastness tests study that better solubility results could be obtained in the presence of ultrasonic energy, all dye solutions were prepared by being stirred for 30 minutes at 50–55ºC in an ultrasonic bath (Baysonic Ultrasound).

Firstly, solutions were prepared with 5 different dispersing agent concentrations (DENPOL HT (Denge Kimya)) at pH 7, with 6 g/l dye concentration. After the determination of optimum dispersing agent concentration, solutions were prepared at 5 different pH values, with 1 g/l dispersing agent concentration at 6 g/l dye concentration. After the determination of optimum pH, dye solutions were prepared at 5 different dye concentrations. Then absorbance values of all solutions were measured at maximum absorbance wavelength (nm) with Jenway type spectral photometer, also photographs of prepared solutions were taken. Furthermore, amounts of dissolved residue were determined with filtration.

Investigation of the usability of synthesized dyes in wool dyeing

To determine the usability of synthesized metal cyclohexane di carboxylate compounds in wool dyeing, the dyeing profile given in figure 1 was carried out at pH 7. The liquor ratio was 1:20 and equalizing and dispersing agent concentrations were 1 and 0.5 g/l, respectively. Dyeing liquors were prepared in an ultrasonic bath, and then dyeing procedures were performed on Thermal HT Dyeing Machine [14].

Since fastness is very important and must be at a sufficient level in textile dyeing, washing fastness tests of dyed samples were performed on James H. Heal 815 20 LGyrowash according to ISO 150 C06 standard. Water fastnesses of the samples were performed according to ISO 105 E01 standard. Acidic and basic perspiration fastnesses of the samples were performed on Prowhite Perspiration Fastness Device according to ISO 105 E04 standard. Dry and

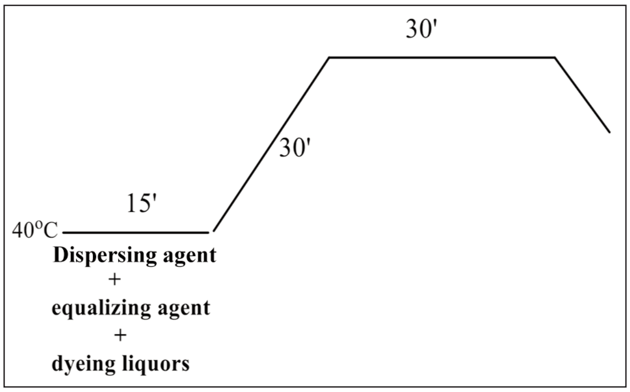

Fig. 1. The dyeing profile

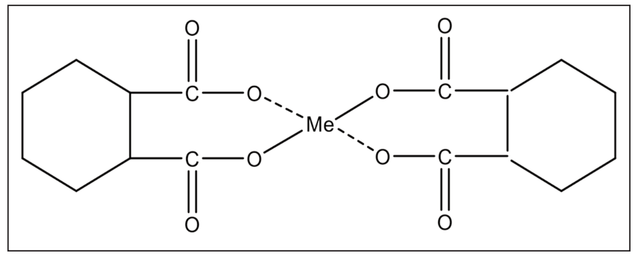

Fig. 2. Molecular structure of metal cyclohexane di carboxylate dye

wet rubbing fastnesses of the samples were performed on Prowhite Manual Crockmeter Device according to ISO 105X12 standard, and light fastnesses of the samples were performed according to 105-B02:2001 ISO 150 C06 standard.

A lack of harmful effects of the dye on human health is important as well as fastness properties. Therefore, to determine the allergic effect of dye, allergy tests were performed on dyed samples with high-performance liquid chromatography & mass spectroscopy according to IHTM AL.2.090&AL.2.178 DIN 54231 standard. Furthermore, to determine the carcinogenic effect of dye, carcinogenic tests were performed on dyed samples with high-performance liquid chromatography devices according to IHTM AL.2.091 DIN 54231 standard in Intertek Testing Laboratory.

RESULTS AND DISCUSSION

Determination of the optimum dissolution conditions for synthesized dyes

Effect of dispersing agent concentration

As determined in our previous study, the dye was not soluble directly in aqueous medium, but dissolved in organic solvents (such as benzyl alcohol). By taking into consideration the molecular structure of the dye that is given in figure 2, it was expected for the compound to have limited solubility in water, because it does not contain groups such as sulpho ($-SO_3^-$) which provides solubility [8].

Production in an organic solvent medium would cause problems in the textile dyeing process in terms of ecological criteria. Therefore, using possibility of the aforementioned dye dispersed in an aqueous medium was investigated. For that purpose, solutions of synthesized dye were prepared with 5 different dispersing agent concentrations (0.5; 1; 1.5; 2; 2.5 g/l) in the presence of ultrasound at 6 g/l concentration and pH 7. Images of filtration sheets of dye solutions prepared in the presence of ultrasound with these 5 different concentrations are given in figure 3. To determine the optimum concentration for dispersing agent, a filtration process was performed to determine whether undissolved dye remained. The solutions were prepared in the presence of ultrasound and passed through filtration sheets. After the filtration, photographs of the filtration sheets were taken. Results are given in figures 3, 4 and 5.

When figures 4 and 5 were examined, it could be seen that cobalt and nickel naphthenates were dissolved completely in all dispersing agent concentrations. Therefore, the optimum dispersing agent concentration was determined to be 0.5 g/l. Although this brought forth the idea that dispersing agents could be

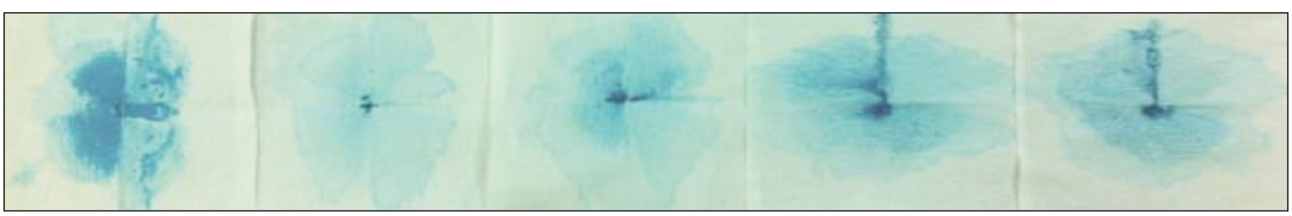

Fig. 3. Images of filtration sheets of dye solutions prepared in the presence of ultrasound with 5 different dispersing agent concentrations (0.5; 1; 1.5; 2; 2.5 g/l) at 6 g/l copper cyclohexane di carboxylate concentration and pH 7

Fig. 4. Images of filtration sheets of dye solutions prepared in the presence of ultrasound with 5 different dispersing agent concentrations (0.5; 1; 1.5; 2; 2.5 g/l) at 6 g/l cobalt cyclohexane di carboxylate concentration and pH 7

Fig. 5. Images of filtration sheets of dye solutions prepared in the presence of ultrasound with 5 different dispersing agent concentrations (0.5; 1; 1.5; 2; 2.5 g/l) at 6 g/l nickel cyclohexane di carboxylate concentration and pH 7

used at lower concentrations, lower concentrations were not studied because it wouldn’t be convenient to work at quite low concentrations in large-scale production conditions.

Effect of pH

To determine in which type of medium (acidic, neutral, basic) the best water solubility of the synthesized dye will be obtained, dye solutions were prepared at 5 different pH values (1; 4; 7; 10; 13) in the presence of ultrasound, at 6 g/l dye concentration containing 1 g/l dispersing agent. To observe whether undissolved dye remained, solutions prepared in mediums with and without ultrasound were passed through filtration sheets, afterwards photographs of the filtration sheets were taken. Results are given in figures 6, 7 and 8.

As a result of the evaluations, it was found that the colour of the dye changed in a strong acidic (pH 1) or strong basic (pH 13) medium. There was not a significant difference in terms of the dye solubility between pH 4, 7, and 10. It was seen that copper, cobalt and nickel cyclohexane di carboxylate compounds could be used in dyeing both acidic and basic environments depending on the fibre type. Furthermore, pH 7 could be considered as the optimum pH for solubility, due to the non-requirement of pH adjustment.

Effect of dye concentration

To determine the maximum solubility of the synthesized dye in water, solutions were prepared at 5 different concentrations (2; 4; 6; 8; 10 g/l) in the presence of ultrasound at pH 7 with 1 g/L dispersing agent and the colours of the solutions were evaluated visually. Images of filtration sheets of copper cyclohexane dicarboxylate solutions prepared in a medium with ultrasound at 5 different concentrations are given in figure 9. Similarly, in this experimental group, to observe whether undissolved dye remained, solutions prepared in the presence of ultrasound, were passed through filtration sheets, afterwards photographs of the filtration sheets were taken. Results are given in figures 9, 10 and 11.

Fig. 6. Images of filtration sheets of dye solutions prepared in medium without ultrasound at 5 different pH (pH 1; 4; 7; 10; 13) at 6 g/l copper cyclohexane di carboxylate concentration with 1 g/l dispersing agent

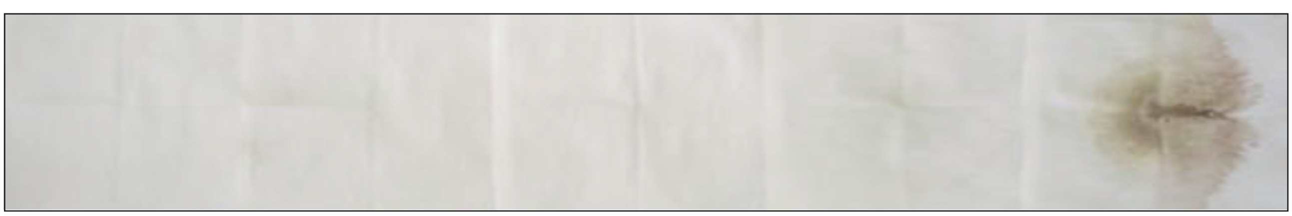

Fig. 7. Images of filtration sheets of dye solutions prepared in medium without ultrasound at 5 different pH (pH 1; 4; 7; 10; 13) at 6 g/l cobalt cyclohexane di carboxylate concentration with 1 g/l dispersing agent

Fig. 8. Images of filtration sheets of dye solutions prepared in medium without ultrasound at 5 different pH (pH 1; 4; 7; 10; 13) at 6 g/l nickel cyclohexane di carboxylate concentration with 1 g/l dispersing agent

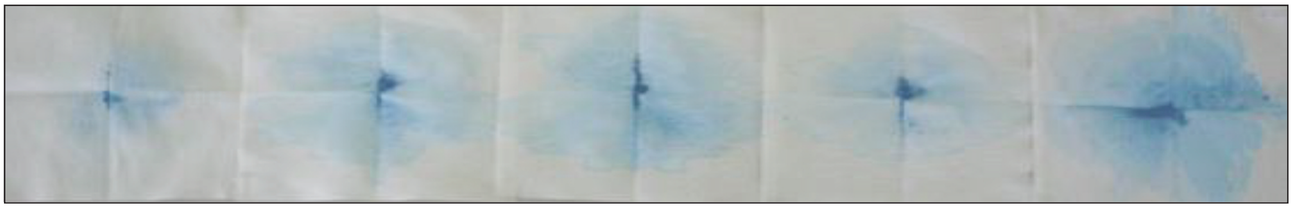

Fig. 9. Images of filtration sheets of copper cyclohexane dicarboxylate solutions prepared in medium with ultrasound at 5 different concentrations (2; 4; 6; 8; 10 g/l) at pH 7 with 1 g/l dispersing agent

Fig. 10. Images of filtration sheets of cobalt cyclohexane dicarboxylate solutions prepared in medium without\ ultrasound at 5 different concentrations (2; 4; 6; 8; 10 g/l) at pH 7 with 1 g/l dispersing agent

Fig. 11. Images of filtration sheets of nickel cyclohexane dicarboxylate solutions prepared in medium without ultrasound at 5 different concentrations (2; 4; 6; 8; 10 g/l) at pH 7 with 1 g/l dispersing agent

When all filter papers were examined, it could be seen that filter papers which contained cobalt and nickel naphthenates were dissolved completely in all dyestuff concentrations. It is of vital importance in textile dyeing, and it is required for dye to dissolve at high concentrations to get dark tones.

Results related to the usability of synthesized dyes in the dyeing of wool fibres

Wool fabric dyed with copper cyclohexane dicarboxylate compound can be seen in figure 12.

Fig. 12. Wool fabrics dyed with copper cyclohexane di carboxylate at 6 g/l dye concentration

From figure 12, it can be seen that the green colour was obtained in wool with copper cyclohexane dicarboxylate.

From figure 13, it can be seen that the red and yellow colours were obtained in wool with cobalt and nickel cyclohexane di carboxylates, respectively. The results of K/S and dCIELab are given in tables 1 and 2.

The results of washing, water and perspiration (acidic and basic) fastnesses of dyed samples are given in table 3

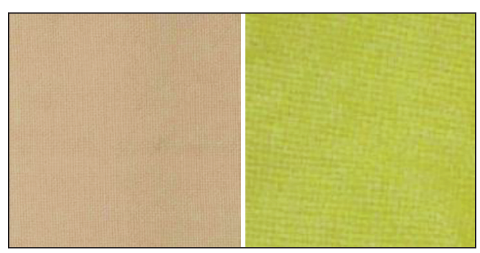

Fig. 13. Wool fabrics dyed with cobalt (on the left) and nickel (on the right) cyclohexane di carboxylates at 10 g/l dye concentration

Table 1

RESULTS OF K/S VALUES METAL CYCLOHEXANE DI CARBOXYLATE DYE

The concentration of metal cyclohexane di carboxylate dye(g/l)	Copper cyclohexane dicarboxylate	Cobalt cyclohexane dicarboxylate	Nickel cyclohexane dicarboxylate
2	3.60	1.73	1.60
6	4.48	1.90	1.91
10	5.61	2.53	2.02

Table 2

RESULTS OF dCIELab VALUES METAL CYCLOHEXANE DI CARBOXYLATE DYE					
Metal Cyclohexane dicarboxylate dyed under optimum conditions	**L***	**a***	**b***	**C***	**h°**
Copper cyclohexane di carboxylate dye	66.12	–1.71	18.66	18.74	95.23
Cobalt cyclohexane di carboxylate dye	65.43	7.06	9.34	11.71	52.91
Nickel cyclohexane di carboxylate dye	71.94	1.97	19.14	19.24	84.11

Table 3

RESULTS OF WASHING, WATER AND PERSPIRATION (ACIDIC AND BASIC) FASTNESSES OF COPPER, COBALT, NICKEL CYCLOHEXANE DICARBOXYLATE DYE							
	Property	**Wo**	**PAC**	**PES**	**PA**	**Co**	**CA**
Copper cyclohexane di carboxylate dye	Washing Fastness	5	5	5	5	5	5
	Water Fastness	5	5	5	5	5	5
	Acidic Perspiration Fastness	5	5	5	5	5	5
	Basic Perspiration Fastness	5	5	5	5	5	5
Cobalt cyclohexane di carboxylate dye	Washing Fastness	5	5	5	5	5	5
	Water Fastness	5	5	5	5	5	5
	Acidic Perspiration Fastness	5	5	5	5	5	5
	Basic Perspiration Fastness	5	5	5	5	5	5
Nickel cyclohexane di carboxylate dye	Washing Fastness	5	5	5	5	5	5
	Water Fastness	5	5	5	5	5	5
	Acidic Perspiration Fastness	5	5	5	5	5	5
	Basic Perspiration Fastness	5	5	5	5	5	5

The results of dry and wet rubbing fastnesses and light fastnesses are given in table 4.

Table 4

RESULTS OF DRY AND WET RUBBING AND LIGHT FASTNESSES			
Dye	**Rubbing fastness**		**Light fastness**
	Dry	**Wet**	
Copper cyclohexane di carboxylate	5	5	4
Cobalt cyclohexane di carboxylate	5	5	3–4
Nickel cyclohexane di carboxylate	5	5	4

Due to the molecular structure of copper, cobalt and nickel cyclohexane di carboxylate dyes, it can be said that the metal atoms, whose coordination number is 4, form two bonds with the dye molecule and have got the opportunity to form two more bonds. These bonds will be formed with water molecules in aqueous medium. In the case of the presence of wool fibre in the environment, it can be bound to the free amino groups of wool fibre with coordinative bonds (figure 14). By taking into consideration that the most robust bond between textile dye and fibres is a coordinative bond, the obtained high wet fastness values could be well understood.

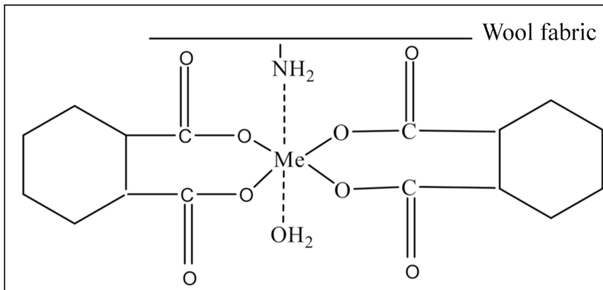

Fig. 14. Possible binding mechanism of metal cyclohexane di carboxylates to wool fibre

As generally known, the light fastness value depends on the dye chromophore. Although copper, cobalt and nickel cyclohexane di carboxylate showed medium light fastness values, by taking into consideration that wool products are not used in warm and hot climates, it can be said that the light fastness value of a dyed woollen material is not critical.

To determine the carcinogenic and/or allergic effect of dye, the aforementioned tests were carried out. According to the allergic test, it can be said that dyes did not cause any allergic reaction to the skin, since the obtained value was below the lowest measurable value, 3 ppm. According to the carcinogenic test, it can be said that copper, cobalt and nickel cyclohexane di carboxylates did not cause any carcinogenic effect, since the obtained value was below the lowest measurable value, 2 ppm.

CONCLUSIONS

The study aimed to examine naphthenates (cyclohexane mono carboxylate) which were obtained from side fractions of petrol and brought in as a new dyestuff to the textile sector. For this aim, different metal naphthenates were synthesized first.

Maximum solubility of copper, cobalt and nickel naphthenate solutions was provided in an ultrasonic environment. Experimentations indicated that three dyestuffs could be used successfully in dyeing wool fibres. Therefore, a new colour gamut, which could be used in wool dyeing and was completely obtained via recycling from waste, was introduced. However, it can be said that the mentioned colour gamut could be used in dyeing only light and medium shades to have high colour fastness.

It should be considered that recycling waste and using it in various areas are going to be more important in the future when the restrictions increase. It can be concluded that the colour gamut synthesized in this study is rewarding under today's conditions where environmental consciousness is increasing.

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