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Metrological Evaluation of Prepared Turbidity Working Standard Solutions to Achieve Traceability for Textile Waste Water Effluents

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WORKING standard must be stable, thus the material should be tested for stability and homogeneity testing complying ISO standard methods with detailed description of how their features and characteristics facilitate all aspects of good laboratory practice (GLP). Stock solutions of (4000) NTU formazin turbidity suspensions were prepared, from this stock a series of diluted suspensions were prepared, and then verified by testing their pH, and spectrophotmatric absorbance values in accordance with published international models. Finally the statistical parameters and accompanied uncertainty for each certified value was calculated. The prepared stock working standard (4000 NTU) solutions were verified via applying the requirements of ISO/IEC 17034 for the homogeneity and stability testing. The results obtained verified the homogeneity and the stability of these working standards as a primary stock for about one year shelf time stability. These prepared stock solutions can be used as working standards for calibrations of turbidity meters that used for testing the turbidity of textile waste water effluents. The main goal of the study is the developing of these standards locally in Egypt. As an applied part the turbidity values of the scouring solutions from different textile fabrics -were measured, these fabrics were ; cotton, wool, silk, nylon and polyester, after calibration of the used turbiditmeter once by the purchased formazin standards and another time by the prepared working standards. Where comparable results were obtained which ensured the suitability of these prepared working standards.

Keywords:Formazin Working Standard, CRM, Homogeneity, Stability, Statistical parameters, Turbidity,Textile effluents, Fabrics,Scouring

Introduction

In textile industry, the textile wet processes considered among the most unfriendly environmentally processes, as these processes produce colored waste-water that is largely polluted with unhealthy materials like: dyes, textile auxiliaries and chemicals. Thus large quantities of waste-water which contains large amount of suspended solids, dissolved organic matters, dyes and heavy metal traces are produced which are capable of harming the environment upon discharge into sewer or municipal water [1-3]. The production processes of textile fabrics involve: scouring, sizing, weaving, desizing, bleaching, mercerizing, dyeing and finishing. Aqueous scouring is the preferred way of scouring fabrics because water considered nonflammable, non-toxic, and cheap [4].

Simply the scouring is a process in which the water insoluble and immiscible materials are converted to water soluble products in saponification process, with a simple reaction between oil and alkali in water to give soap and glycerin .Thus the components in the scouring bath should be selected with respect to the fiber type in mind, as in case of cotton scouring strong alkali can be used, but wool and rayon are damaged by it. Wool can withstand acids whereas cotton, rayon and nylon cannot [5].

Turbidity comes from the Greek word (turbid) means cloudy, hazy or not pure due to dispersed suspended solids such as silt, clay, algae, organic/inorganic matter or microorganisms [6]. Turbidity can impact water quality with subsequent health and cleanliness fallout, this fact making turbidity to be one of the many water quality criteria.

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Simply turbidity can be defined as a decrease in the transparency of a solution due to the presence suspended and some dissolved substances, which causes incident light to be scattered, reflected and attenuated rather than transmitted in straight lines : the higher the intensity of the scattered or attenuated light , the higher the value of turbidity [7,8].Turbidity can be also expressed in nephelometric turbidity units (NTU) which can be defined as the intensity of light- at a specified wave length- scattered or attenuated from the bath of the incident light by suspended particles at a specified angle usually 90 degrees.

In accordance with the agencies for environmental reorganization such as the American Public Health Association, the American Water Works Association and the U.S. Environmental Protection Agency formazin turbidity standard was accepted as the primary standard in methods of turbidity testing for the chemical examination of water and wastes. Where, formazin is an aqueous suspension of an insoluble white polymer [9] formed by the condensation reaction between hydrazin sulfate and hexamethylenetetramine. The preparation, storage, and handling of formazin will affect its accuracy and stability [10]. Working standards are prepared by volumetric dilution of 4000-NTU stock formazin with deionized water. Formazin standards are also the common standards for the calibration of turbidimeters according to standard method [11]. Such working standards are used to check instrument calibration stability.

The present study dealt with the characterization of the prepared turbidity solutions, where a series of six turbidity samples were prepared from the prepared stock of 4000 NTU and the work continued with detailed description of the fulfillment of the requirements of ISO guides standard for stability and homogeneity of such prepared suspensions. Finally as an applied part we measured the turbidity values of the scouring solutions of different textile fabrics: cotton, wool, silk, nylon and polyester after calibration of the used turbiditmeter once by the purchased formazin standards and another time by the prepared working standards to ensure the suitability of these prepared working standards.

Experimental Work

Chemicals

1. Ultra-low turbidity deionized water of high quality for the preparation of the formazin calibration suspensions.
2. Hydrazine sulfate ($N_2H_6SO_4$): analytical reagent, of molar mass 130.12 g^{-1} $mol^{-1}$and purity 99%.
3. Hexamine ($C_6H_{12}N_4$): analytical reagent, of molar mass 140.186 g^{-1} mol^{-1} and purity shall be greater than 99%.

These chemicals were supplied by Trading Dynamic Company, El Haram- Giza- Egypt.

4. Purchased standard turbidity solutions. Supplied by HACH company , Loverland CO, USA.

Devices and instruments

-Three turbidimeters were used in testing of turbidity values, device no.1 of: model 2100N, device no.2 of: model HI- 89703, and device no.3 of: model WTW Turb 550.

- Electronic balance. Model BP 221 S, made by Sartorius, Germany.

-pH –meter model pH -900 made by Swiss Mode.

-A Varian (UV-VIS-NIR) spectrophotometer.

All of the instruments and glassware used were calibrated to achieve the traceability to SI measurement system.

Preparation of formazin standard turbidity solution

Formazin standard turbidity solutions can be prepared in accordance with standard test method [11- 13] as shown in the following scheme:

$$C_6H_{12}N_4 + 6H_2O + 2H_2SO_4 \rightarrow 6H\text{-}CHO + 2(NH_4)_2SO_4 \text{------------------------} (1)$$

Hexamethylene tetramine (from hydrazine sulfate) Formaldehyde Ammonium sulfate

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$$nH\text{-}CHO + n/2: NH_2\text{---}:NH_2 \rightarrow [C_{17}H_{13}N_5O_3]_x + nH_2O \text{----------------------} (2)$$

Formaldehyde Hydrazine Formazin

Scheme of Formazin Formation

where the stock solution of turbidity value (4000) NTU was prepared as stated in the following steps:

- A 5.0 g of hexamethylenetetramine ($C_6H_{12}N_4$) was dissolved in approximately 40 ml of water in a graduated beaker marked as: reagent 1 and 0.5 g of hydrazine sulfate ($N_2H_6SO_4$) in approximately 40 ml of water in another graduated beaker marked as: reagent 2 separately then pour each of the prepared reagents (1&2) into a 100.0 ml volumetric flask, dilute to the mark with ultra-low turbidity water and mix well. Leave each volumetric flask for 24 hr at 25^o C in the dark.

-Then 10 ml of reagent 1 and 10 ml of reagent 2 were mixed in a black volumetric bottle closed well and stored for 48 hr in a dark place. After the specified time a white formazin suspension having a value (4000NTU) was obtained which will be considered as the stock solution that will be used in the preparations of a series of diluted turbidity suspensions.

-Care should be exercised with the formazin standards as these standards have high settling rates [10] and should be remixed at least every 15 min before measurements.

From the prepared stock formazin of (4000NTU) solution a series of diluted suspensions were prepared which include (10, 20, 100, 500, 750 and 1000) NTU by the dilution equation:

Formula of preparation: A= K x B/C

where: A: volume (ml) of stock liquor (4000 NTU), B: solution strength required (NTU), C: Strength of stock formazin (4000 NTU), and K: Total preparing volume (ml), in accordance with the following calculated analytical dilutions (Table 1):

Measurement of the turbidity values of the prepared solutions

The turbidity values of the prepared stock solutions 4000 NTU and the diluted prepared series of (10, 20, 100, 500,750, and 1000 NTU) were measured replicated for five time readings on three calibrated turbidimeters in accordance with standard test method [9].

Measurement of the pH values of the prepared solutions

The pH values of prepared turbidity solutions(10,20,100, 500,750, 1000 and 4000 NTU) respectively were measured each for five time readings on a pH -meter which has been calibrated in accordance with standard test method [14].

Measurement of the absorbance values of the prepared solutions

The absorbance values of all of the prepared turbidity solutions (10, 20,100, 500,750, 1000 and 4000 NTU) were measured using a Shimadzu UV/Visible spectrophotometer measurements to verify the turbidity values of the prepared solutions from absorption data in accordance with Kevin L.Goodner model [15]. This model is presented to obtain turbidity information from the absorption of light at 750nm. The model is a simple linear model with r^2=0.98 and 95% prediction probability.

Calculation of statistical parameters and uncertainty estimations of the prepared working standard solutions

Statistical parameters which involve: mean value, standard devision, variance, expanded uncertainty, and bias of all measured turbidity values of all the prepared turbidity solutions (10, 20,100, 500,750, 1000 and 4000 NTU) were calculated from excel functions according to ISO Guide 35[16].

TABLE 1. The calculated analytical dilutions prepared from 4000 NTU Stock

Required (NTU) Standards	Volume (ml) of (4000 NTU) Stock solution
10	0.25
20	0.50
100	2.50
500	12.5
750	18.75
1000	25.0

* The preparations were carried out in 100 ml measuring flasks.

Verification of the prepared stock working standard (4000 NTU) solution

Verification of the prepared stock standard (4000 NTU) solution was done as an essential to prepare the other suspensions of turbidity levels within the range of interest to fulfill the requirements of ISO Guide 34 where the homogeneity and stability testing of the prepared stock standard were performed [17,18].

Applied part

Measuring of turbidity values of the scouring solution of different textile fabrics

Fabrics:

1-Cotton fabrics were of 150 g/m^2 supplied by El-Shorbagy Company Egypt.
2-Wool fabrics with a weight 130 g/m^2 supplied by Golden Tex Company, Egypt,
3-Silk fabrics were of 69.83g/m^2 supplied by El-Khateeb Factory, Akhmeem, Egypt.
4-Polyester fabrics were of 108 g/m^2 supplied by 'Ouf Son's' Company, Egypt.
5-Nylon fabrics with a weight of 0.9963 g/m^2 supplied by El-Shorbagy Company Egypt.

Scouring process of different textile fabrics:

Scouring was carried out for five types of textile fabrics those are cotton ,wool, silk, polyester, and nylon using 2g/l nonionic detergent and 2g/l sodium carbonate with L.R. 1: 50 at temperature 40^{o}C for 30 min according to standard test method [19] of commercial scouring. Then the scoured fabrics were rinsed with distilled water. Then we measured the turbidity values of the scouring solution from different textile fabrics using turbidity-meter after its calibration once by the purchased formazin standard and another time by the prepared formazin working standards to ensure the suitability of the prepared formazin working standards.

Results and Discussion

Results of the turbidity values of the prepared solutions

Tables 2-4 showed the results of the measured turbidity values of the prepared solutions suspensions of turbidity levels within the range of interest (10, 20, 100, 500,750, 1000, and 4000) NTU using three turbidimeters to verify their turbidity values. The measurements are monitored through repeating the turbidity measurements three times along six months, where the prepared suspensions are stored at about 20^{o}C in brown bottles in a dark place. The obtained results approved approximately stable values along the studied range especially for higher turbidity values [20]. However, formazin standard at a concentration of 4000 NTU maintains its turbidity value for about one year, where residual reactants and reaction intermediates (largely hexamethylenetetramine) compose the polymer's matrix. While dilutions with ultra-low turbidity water, which dilute the matrix of the formazin appears to aid in the degradation of the polymer. This degradation may be caused by coagulation that occurs from the hydrolysis of the formazin polymer [21].

TABLE 2. Turbidity values of the suspension solutions measured on January/2015 and measured using different turbidimeters

Working standard of (NTU) values	Individual Measured (NTU) values of:		
	Device No.1	Device No.2:	Device No.3
>0.1	0.02	0.03	0.02
10	10.1	9.9	9..8
20	20.0	20.1	19.9
100	97.1	97.5	97
500	495	494	495
750	746	749	750
1000	1001	1000	1000
4000	4002	4001	4001

Device No.1 of: Model 2100N turbidimeter , Device No.2 of: Model HI- 89703, and Device No.3 of: Model WTW Turb 550,were calibrated using the purchased turbidity standards supplied with each device

TABLE 3. Turbidity values of the suspension solutions measured on April/2015 and measured using different turbidimeters

Working standard of (NTU) values=	Individual Measured (NTU) values of:		
	Device No.1	Device No.2:	Device No.3
>0.1	0.03	0.03	0.03
10	10.2	10	10
20	20.1	20.2	20.3
100	97.4	97.5	97
500	493	497	495
750	746	748	750
1000	1001	1001	1000
4000	4002	4001	4001

TABLE 4. Turbidity values of the suspension solutions measured on July/15 and measured using different turbidimeters

Working standard of (NTU) values=	Individual Measured (NTU) values of:		
	Device No.1	Device No.2:	Device No.3
>0.1	0.05	0.04	0.04
10	10.3	10.1	9.9
20	20.2	20.3	19.5
100	97.5	97.5	97.4
500	495	497	495
750	745	748	750
1000	1003	1003	1001
4000	4009	4010	4013

Measurement of the pH values of the prepared solutions

The results of the pH values of the prepared turbidity solutions (10, 20,100, 500,750, 1000 and 4000 NTU) respectively are listed in (Table 5) .The results showed that the pH varied within the range (8.02-8.77) which confirming the basic nature of the prepared formazin suspensions in accordance with the range (8-11) that stated in literature [22]. Where the main chemical reagent in the prepared formazin polymer was the hexamine which is a weak base and will result in making the composition basic, thus, the more the basicity, i.e., higher pH values means more stable will be the suspension. These results verified that these working standards can be used for the calibration of turbidimeters used for examination of textile waste water in accordance with standards method [23].

TABLE 5. The pH values of all of the prepared turbidity solutions

Standard (NTU) values =working standard	Average pH values
>0.1	8.04
10	8.02
20	8.05
100	8.06
500	8.42
750	8.30
1000	8.47
4000	8.77

Measurement of the absorbance values of the prepared solutions

Figures 1&2 show the full absorbance spectra for all of the prepared turbidity solutions (10, 20,100, 500,750, 1000 and 4000 NTU) that measured at range from 400-790 nm .The estimated turbidity values of the prepared solutions were calculated using Kevin L.Goodner model [15] which is presented to obtain turbidity information from the absorption of light at 750 nm. The model is a simple linear model with r^2=0.98 and 95% prediction probability, where the prediction probability lines are used to estimate the error, for example, if we measured the absorbance at 750 nm to be 0.200 then the estimated NTU would be approximately 185 NTU. The estimated turbidity values calculated using L.Goodner Model were given in Table 6.These results verify the turbidity values of those prepared turbidity solutions.

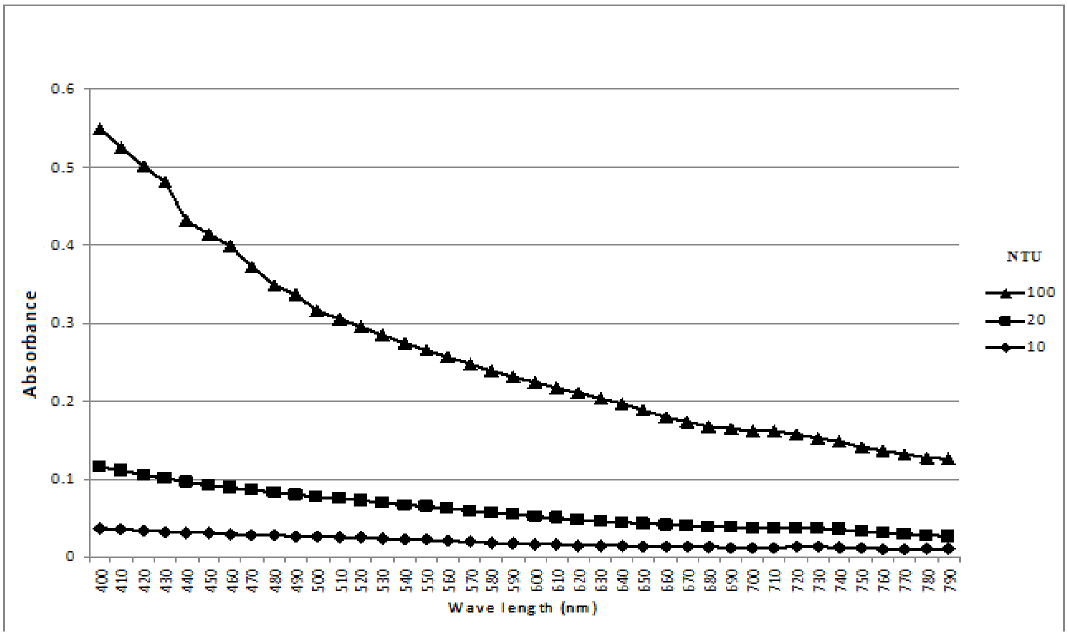

Fig. 1. The absorbance spectra for all of the prepared turbidity solutions (10, 20, and 100 NTU) measured at 750 nm.

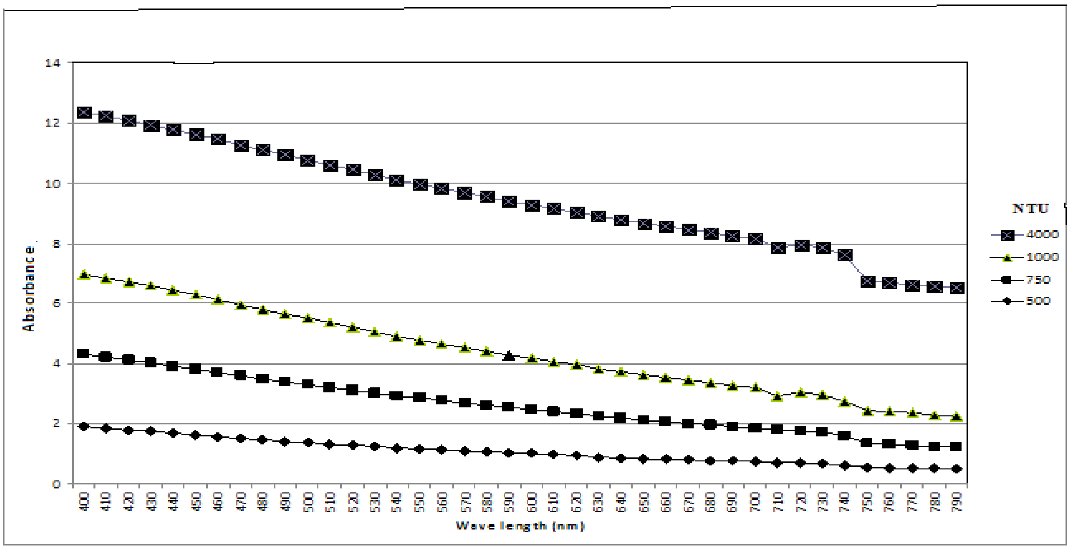

Fig. 2. The absorbance spectra for all of the prepared turbidity solutions (500, 750, 1000 and 4000 NTU) measured at 750 nm.

TABLE 6. Absorbance values of all of the prepared turbidity solutions and their estimated (NTU) values.

Standard (NTU) values	Absorbance values At 750nm	Estimated (NTU) values From Golden Model
10	0.0107	9.95
20	0.0216	19.98
100	0.108	99.90
500	0.540	499.50
750	0.810	749.25
1000	1.081	999.93
4000	4.32	3996.0

Statistical parameters and uncertainty estimations of the prepared working standard solutions

Table 7 represented the statistical parameters and uncertainty values of the prepared working standard turbidity solutions calculated for each of them using the excel functions according to standard method [24].Where statistical parameters involved standard deviation, variance, uncertainty type A, expanded uncertainty, Bias calculation. The uncertainty is a parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand [24] , where a measurand is defined as a particular quantity subject to measurement .The components involved in the calculation of uncertainty budget should be quantified and were determined in accordance with the Ishikawa diagram that known as a 'fishbone' diagram which illustrating these parameters . These components are uncertainty of standard (U_{stand}) which is related to purity of chemicals used in their preparation , uncertainty of the used balance ($U_{balance}$) , type A uncertainty related to the repeability of measurement ($U_{repeatability}$), uncertainty of resolution of the used turbidimeter ($U_{resolution}$) .The combination of all of these uncertainties give the combined uncertainty of the prepared standard ($U_{Combined}$), from which we calculated the expanded uncertainty ($U_{Expanded}$) using the relation :

$$(U_{Expanded})=2x\ (U_{Combined})$$

Expanded uncertainty was used in order to obtain uncertainty results corresponding to confidence level 95%.

The results showed that the calculated expanded uncertainty varied with turbidity value, where the smaller the uncertainty values the better the accepted variation of the tested measurand.

The bias values were calculated for each formazin suspension in order to establish that uniform procedures for calculating and reporting measurements system data quality indicators are used where a bias is a systematic error of a measured value and is given by ;

Bias = Average value – True vale /True value.

The results showed that the bias may be negative or positive and is expressed in the units of measurements or as a percentage of the value of the standard, where a positive bias implies that too high results are obtained.

Verification of the prepared stock standard (4000 NTU) solution

Verification of the prepared stock standard (4000 NTU) solution was done as an essential to prepare the other suspensions of turbidity levels within the range of interest to fulfill the requirements of ISO Guide 17034 for a matter to behave as a working standard where the homogeneity and stability testing of the prepared stock standard were performed [18].

Stability testing: stability of prepared working standard (4000 NTU) along 12 months

Stability testing is performed according to ISO Guide 35 or even surpasses the recommendations given therein [16].The results recorded in Table 8 showed the turbidity values of working standard (4000NTU) measured using different turbidity-meters along 12 months. The variations in the obtained readings were analyzed by applying statistical approach for the control chart of one of the used three calibrated turbidity-meters in order to verify that variation lied between the upper and the lower warning limits of variation which is the safe region, were represented in figure (3).

Stability monitoring [14, 16]

Verification of the prepared stock standard (4000 NTU) solution was done as essential to prepare the other suspensions of turbidity levels within the range of interest to fulfill the requirements of ISO Guide 34 [18] where the homogeneity and stability testing of the prepared stock standard were performed.

Our work concerned with long-term stability which is defined as the stability of a property of a reference material under specified storage

TABLE 7. statistical parameters and uncertainty values of the prepared working Standard turbidity solutions.

Standard Working Solution NTU	Average measured turbidity values	Standard deviation	Variance	Uncertainty type A	Expanded Uncertainty ± NTU	Bias (NTU)
>0.1	0.3111	0.01	0.0	0.00	0.00039	0.20
10	10.70	0.01	0.1	0.04	0.0147	0.70
20	20.50	0.01	0.01	0.04	0.369	0.25
100	96.68	0.13	0.13	0.36	0.015	0.50
500	496.65	0.86	0.93	0.35	0.0127	-3.32
750	748.0	0.84	0.7	0.32	0.0126	-2.06
1000	1001.43	0.80	0.89	0.34	0.0127	1.43
4000	4001.38	1.24	1.11	0.42	0.0126	1.38

*Expanded uncertainty was calculated with respect to confidence level 95%.

TABLE 8. Turbidity values of working standard (4000NTU) Measured using different turbidimeters along 12 months.

Months	Individual Measured (NTU) values of:		
	Device No.1	Device No.2:	Device No.3
January	4000	4001	4001
February	4001	4001	4001
March	4001	4002	4001
April,	4001	4002	4001
May,	4008	4002	4010
June,	4009	4005	4010
July,	4009	4005	4010
August	4010	4005	4010
September	4010	4005	4010
October	4015	4005	4012
November	4015	4005	4012
December	4018	4005	4016

Device No.1 of: Model 2100N, Device No.2 of: Model HI- 89703, and Device No.3 of: Model WTW Turb 550,

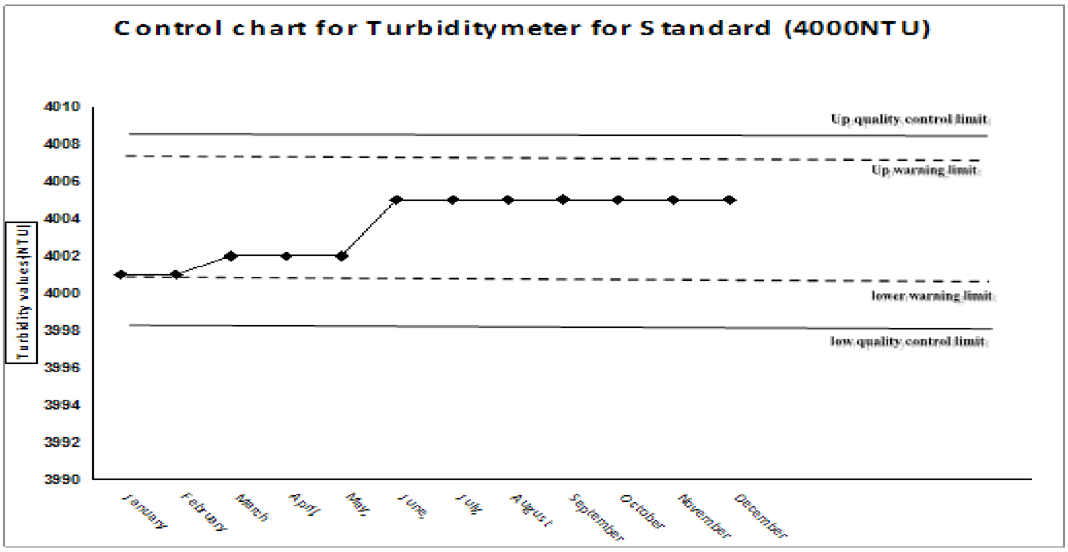

Fig. 3. Control chart for turbidity-meter of model 2100-N using prepared working standard (4000NTU).

conditions at the CRM-producer. And we define the shelf life of a reference material which is defined as the time interval during which the producer of this material warrants its stability and it is equivalent to the period of validity of the certificate, as described in ISO Guides [25].

Where monitoring of stability usually takes place using the classical design in which reference material samples are prepared at the same time under identical conditions and measurements are carried out as time elapses under reproducibility conditions.

Thus the long term stability of the prepared working standard stock 4000NTU were assessed by the analysis of these samples at 6 time points (0, 1,3, 6, 9 and 12 months) at (20 °C). The samples are analyzed at each time point for their mean and standard deviation values where these results are recorded in the Table 9.

Standard deviation is a statistical measurement describes the dispersion of a measured quantity from its mean value and calculated from the square root of the variance, thus the wider the range is means the greater the standard deviation is and the riskier an investment is considered to be. The results obtained showed minimum standard deviation values of the tested working standard which means better stability of the tested samples [18].

TABLE 9. Monitoring of stability of 4000 NTU at 6 time points at (20 °C).

Turbidity values at 20°C	0	1	3	6	9	12
	4000	4000	4001	4014	4024	4025
	4002	4002	4002	4010	4025	4024
	4001	4001	4001	4012	4024	4025
	4001	4001	4001	4012	4022	4024
	4001	4001	4001	4012	4024	4025
Mean	4001	4001.4	4001.6	4012	4023.8	4024.6
(S_{stab})	0.707	0.894	0.548	1.414	1.095	0.548

Mean=$\overline{x}$ = ($X_1 + X_2 + \cdots X_n$) / n , (S_{stab}) standard deviation of stability= [∑ (xi-^{2}($\overline{x}$ / (n-1)]$^{1/2}$,x_i being the result of the i-th measurement and $\overline{x}$ being the arithmetic mean of the n results considered.

Homogeneity testing

In order to perform homogeneity testing the turbidity values of three samples of working standard 4000 NTU were measured five times and the data are shown in the Table 10. The data were examined and discard any values which have been determined to be outliers. Then apply the statistical model ANOVA (Analysis of Variance) [25]. Where ANOVA provides a statistical test of whether or not the means of several groups are equal and applied to determine the between sample and the within sample variability using the Microsoft excel. The results from ANOVA are represented in table (11). In analyzing these results obtained from ANOVA representation for homogeneity criteria we concluded that: the Fisher value calculated value $F_{calculated}$, was smaller than the Fisher value tabulated $F_{critical}$ taken, *i.e.*, $F_{calculated} < F_{critical}$ suggesting the probability value P-value ≥ 0.05. From this conclusion we verified that the prepared working standard (4000 NTU) samples solutions were homogeneous and they match the requirements to be used as a turbidity working standards [26].

TABLE 10. Turbidity values of three samples of working standard 4000NTU for Homogeneity Testing.

Within sample	Run No	Sample Number		
		1	2	3
	1	4001	4000	4001
	2	4001	4001	4001
	3	4002	4001	4001
	4	4001	4001	4001
	5	4002	4000	4000
	Total Sum	20007	20003	20004

TABLE 11. ANOVA table for homogeneity calculations.

Achievement Source of variation	Sum of Squares $SSx10^7$	Degree of freedom (df)	Mean Square (MS)	Calculated Fisher value (F_c)	Tabulated Fisher value (F_{alpha})= ($F_{critical}$)
Between Groups	SSA=2.4084068	K-1=2	MSA=SSA/ K-1 $=MS_{between}$	F_c =MSA/ MSE $F_c = 2x10^{-5}$	(F_{alpha})=3.89 Suggesting Probability value=0.05
Within Groups	SSE=7.2184928	N-k=12	MSE=SSE/K-N $=MS_{within}$		
Total	SST=9.6268996	N-1=14			

where SSA is the sum of squares among samples, SSE is the sum of squares related to errors, SST is the total sum of squares $MS_{between}$ is the sum of squares for the between samples, MS_{within} is the sum of squares within sample, $F_{calculated}$ is the Fisher value calculated, $F_{critical}$ is the Fisher value tabulated, P-value is the probability value.

Results of applied part

The turbidity is an important aspect of textile wastewater thus we measured the turbidity values of the scouring solution from different textile fabrics using turbidity-meter after its calibration once by the purchased formazin standard and another time by the prepared formazin working standards to ensure the suitability of the prepared formazin working standards. The results obtained are represented in Table 12 and the changes are clarified through Fig. 4.The results showed that the order of turbidity values was:

Cotton > wool > silk > nylon > polyester.

Thus the highest turbidity values were recorded for scouring solutions of cotton fabrics this may be due to the fact that [27] cotton fabric is covered with a thin film of cuticle, which plays major role in the surface qualities of the fibre and consists of wax and pectic material along with some mineral matter,all of these non-cellulosic materials may be removed by scouring leading to increased turbidity of their scouring solutions. From this result it can be concluded that turbidity of a scouring solution is related to fabric type and its nature in accordance with some literatures [4, 5].

Also, comparable or approximately the same turbidity values were obtained from the two solutions. From these results we ensured the suitability of the prepared formazin working standards.

TABLE 12. Measured Turbidity values of the scouring solution from different textile fabrics.

Textile Fabrics Names	Average measured turbidity* values after calibration by:	
	Purchased formazin standards	Prepared formazin working standards
Cotton	170.2	169.0
Wool	120.2	120.0
Silk	98.5	98.0
Polyester	63.9	63.3
Nylon	94.2	93.8

*Each measured value was taken as an average of five separate readings for each fabric.

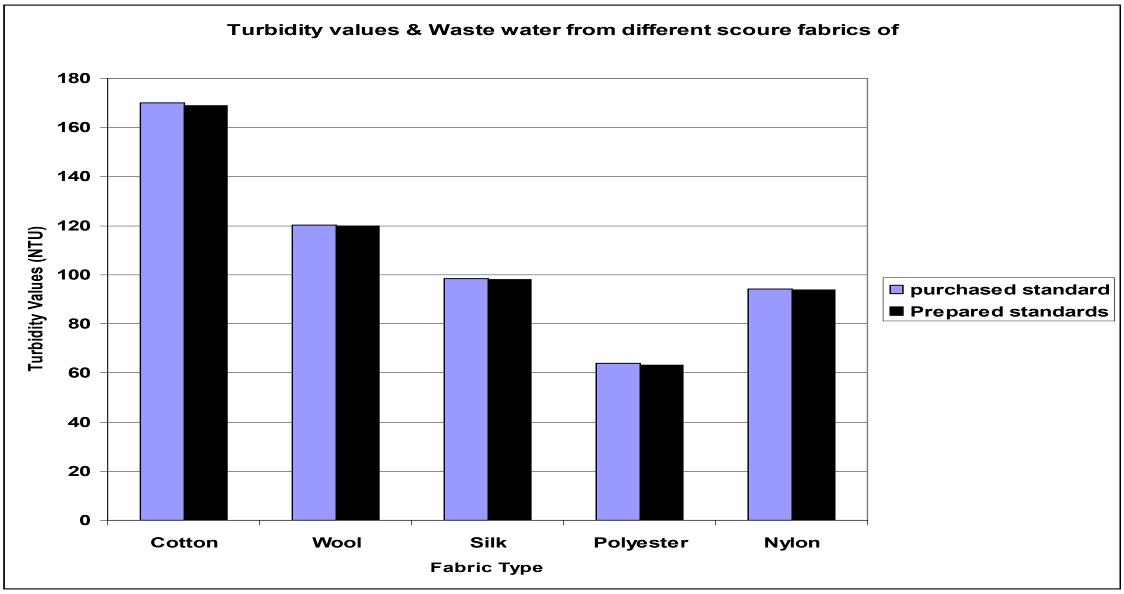

Fig. 4. Turbidity values of the scouring solution from different textile fabrics

Conclusions

- Turbidities of working standards carefully prepared from the last prepared stock formazin (4000NTU) were stable for about six months. The stability of these diluted species depending on their concentration and on how often the settled formazin is re-suspended. While the prepared stock (4000NTU) is stable for about one year. Preparations left undisturbed for long periods tend to be less stable than those which are mixed occasionally.
- The results obtained verified the homogeneity and the stability of the prepared working formazin standards (4000NTU) as a primary stock for one year shelf time stability in accordance with applied ISO standards. Dilute formazin suspensions used as working standards are considerably less stable. As a rule the lower the concentration of a formazin standard, the lower the stability of that standard. These prepared standards serve as working standards in calibrations of turbiditmeters for testing

the turbidity of textile waste water effluents. As regulatory requirements the measurements should be fully validated, traceable to primary reference materials and, most importantly, should be comparable. The production of such traceable working standards is the main goal of the study instead of purchasing them for complying economic recommendation.

- The present work ensured the suitability of the prepared formazin working standards. Where a comparable or approximately the same turbidity values were obtained for the scouring solution from different textile: cotton, wool, silk, nylon and polyester after calibration of the used turbiditmeter once by the purchased formazin standards and another time by the prepared formazin working standards.

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التقييم المترولوجي لمحاليل العكارة العيارية المحضرة لتحقيق الاسناد للنفايات السائلة لمياه الصرف فى صناعة النسيج.

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تعتبر العمليات النسيجية الرطبة في صناعة الغزل والنسيج من بين أكثر العمليات غير الصديقة للبيئة، حيث أن هذه العمليات تنتج مياه صرف ملوثة إلى حد كبير بمواد غير صحية مثل: الأصباغ والمواد الكيميائية. وبالتالي يتم إنتاج كميات كبيرة من الملوثات التي يمكن أن تلحق الضرر بالبيئة عند تصريفها في الصرف الصحي . والعكارة تعتبر واحدا من العديد من معايير نوعية المياه الناتجة من صناعة النسيج.لذلك ارتكز البحث لتحضير محاليل العكارة المستخدمة فى معايرة اجهزة قياس العكارة وهى محاليل قياسية من الفورمازين بمحلول اساسى بقيمة (4000 وحدة عكارة (NTU).

كما تمت دراسة مدى توافق عاملى التجانس (Homogeneity) والثبات (Stability) لهذه المحاليل وانطباقها مع اشتراطات المواصفة القياسية (ايزو17034) لانتاج المواد المرجعية .
وتم استخدم هذا المحلول الاساسى فى تحضر العديد من التركيزات الاقل بدرجات عكارة مختلفة تتراوح بين 10ال 1000 وحدة عكارة(NTU) .

وكجزء تطبيقى للبحث تم اخذ عملية الغسيل القياسى(Scouring) كمثال لواحدة من العمليات التى تتم على الاقمشة النسجية لعدد خمسة انواع من اقمشة: القطن ،الصوف ،الحرير ، النيلون والبولى استر ، ثم قياس درجة العكارة لمحاليل غسيل هذه الاقمشة النسجية باستخدام جهاز قياس العكارة بعد معايرته بطريقتين الاولى باستخدام محاليل فورمازين قياسية تم شراؤها والثانية بعد معايرته باستخدام محاليل فورمازين المحضرة بالمعمل ، واوضحت النتائج مدى ملائمة تلك المحاليل المحضرة حيث اعطت تقريبا نفس القيم لدرجات العكارة من الاجهزة التى تم معايرتها باستخدام المحاليل التى تم شراؤها ، وهذا يوضح اننا استطعنا تحقيق الهدف الاساسى من الدراسة فى هذا البحث وهو استخدام المحاليل القياسية المحضرة بدلا من انفاق الكثير فى شراؤها.