Preparation and Characteristic Study of Pure and Na₂O Doped NiO-Mn₂O₃-MgO Catalyst

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Abstract :

The catalyst NiO-Mn₂O₃-MgO was prepared in the ratio of 10:50:40 for the mixed oxides respectively, by using the coprecipitation method of their mixed bicarbonates, from their nitrates solution using 1M NaHCO₃ solution at constant pH ,dried at 100°C and calcinated at different temperatures (400,500,600,700 and 800) °C for 4 hours . The Na₂O doped mixed oxide were prepared by impregnating a given dry weight of Ni,Mn and Mg basic bicarbonate M (HCO₃)₂ with a solution containing different amounts of sodium nitrate (2%,4%,6% and 8%) sufficient to make paste. The paste were dried at 110°C and then calcined at 600°C for 4 hrs . surface and catalytic properties of the pure and Na₂O doped catalyst were investigated using XRD, atomic absorption spectrophotometer, UV-visible techniques and wet impregnation method. The degree of crystallinity and crystallite size of the produced phase increased progressively as a function of calcination temperature from (400 to 600)°C,but decreased at (700 and 800) °C .The catalytic activity of pure and doped catalyst was tested in the degradation of reactive red dye in the presence of ultraviolet light, and study the effect of temperature on the activity of catalyst. The results showed that the doped catalyst has a smaller activation energy (13 kJmol^{-1}) than the pure catalyst $(17.8 \text{ kJmol}^{-1})$ $kJmol.^{-1}$).

Key words:NiO catayst,Mn₂O₃ supported catalyst,Na₂O doped catalyst

Chemistry Classifiication QD146-197

Introduction:

releasing toxic and potential carcinogenic substances into the aqueous phase, and the No. 1 polluter of clean water(Reife,1996).Because of the complicated molecular structures of dyes make dye wastewater difficult treated by conventional to be biological physico-chemical and process(Kirk and Farrell, 1987). Therefore, heterogeneous the photocatalysis was used as an attractive method of wastewater treatment capable of removing colour and toxic organic compounds from textile effluents, including the dyes,. Heterogeneous photocatalysis is a process in which a combination of photochemistry and catalysis is operable and implies that light and catalyst are necessary to bring out a chemical reaction (Stylidi et al., 2003). Manv

catalysts like ZnO, TiO₂, ZrO₂, WO₃, SrO₂, Fe₂O₃, Nb₂O₅, CeO₂, CdS and ZnS have been attempted for the photocatalytic degradation of a wide variety of environmental contaminants (Balcioglu and Arslan, 1998; Moraes, 1999; Santana, 2002; Kansal and Navjeet,2009; Attia et al.,2008). The effect of addition of Na₂O to the metal oxides supported catalyst lead to the changes on physicochemical, specific surface areas, catalytic Properties, and solid-solid interactions between of different transition metal oxides supported on alumina or such Co₃O₄/MgO magnesia as (Deraz,2001), Mn_2O_3/Al_2O_3 (Deraz,2002), and NiO/Al₂O₃ (El-Shobaky et al., 1985) and consequently on their catalytic activities (Shaheen The aim andSelim,1998).

of this study is to prepare the catalyst

Mixed oxides containing transition metal oxides are used to design the catalytic materials, Manganese oxides are reported to be among the most efficient transition-metal compounds in composition (Álvarez catalyst et al.,2003; Drama et al.,1995; Craciun et al.,2003;Lahousse et al.,1998). Mixing with transition-metal manganese oxides in many catalytic reactions might modify the catalytic activity of components (Li,2003; separate Morales,2006).Mixed oxide solids containing Mn are active catalyst for oxidation-reduction reactions and combustion processes , for example the oxides(Co.Zn.and Mn) were active in reduction the of nitrous oxide(Fierro,2001) ,and Co-Mn oxides were active in the conversion of synthesis gas to light olefins (Mirzaei, 2006) and oxidation of ethanol(Kovanda,2006), Also, Co-Mn oxides and Ag-Mn oxides supported on Al2O3 have been reported to act as catalysts used in oxidation volatile organic compounds (Luo,1998). Dyes are synthetic and aromatic molecular structural compounds .The textile dyes can be classified according to their dissociation in an aqueous solution, and their method of application to (anionic), basic dves dves acid (cationic) and disperse dyes (nonionic), they are used on several substrates in food, cosmetics, paper, plastic and textile industries.(Campos,2001).

Reactive dyes are anionic dyes most commonly applied to cotton, that form covalent bonds with the hydroxyl groups in the cellulose These (Needles, 1986).

textile dyes create severe environmental pollution problems by 2018

that was resulting from textile factories, which is the main pollutant of the soil and water.

Experimental Part:

1-Preparation of Catalyst:

the mixed bicarbonates with calculated amount of sodium nitrate(purity 99% Fluka supplied from Company) dissolved in the least amount of distilled water sufficient to make paste. The paste were dried at 110°C and then calcined at 600 °C for 4 hrs. X ray powder diffractograms of various investigated samples were carried out using a Bruker diffractometer (Bruker D8 advance target). The patterns were with CuK**a**1 with run secondly monochromator($\lambda = 0.1545$ nm) at 40 kV and 40 mA.

minutes and then cooled and weighed (W2),and then weighing it immersed in water(W3).The physical properties calculated by using these weights:

Apparent porosity $(P_A\%) = (W2-W1 / W2-W3) *100$ pore Volume $(V_P) = W2-W1 / W1*d$ d_a : water density particle density $(D_P) = W1 d_a / W2-W3$ NiO-Mn₂O₃-MgO, in the ratio 10:50:40 for the mixed oxide respectively, study of its physical properties .study the effect of Na₂O ratios in its catalytic activity, we used the pure NiO- Mn₂O₃-MgO and Na₂O doped catalyst in the presence of ultraviolet light to remove the reactive red dye from industrial wastewater,

NiO- Mn₂O₃ -MgO mixed oxide prepared coprecipitation by was method of their mixed bicarbonates from their nitrates(purity 99% supplied from Merck and B.D.H.Companies) using NaHCO₃(99% solution 1Msupplied from B.D.H Company) solution at constant pН and temperature 70°C. The carefully washed precipitate was dried at 110°C till constant weight, then formulated as a tablets by diameter 10 mm, and then subjected to heating at 400, 500, 600,700 and 800°C for 4 hrs. Four Na₂O - doped samples were prepared by impregnating a given dry weight of **2-Physical Properties** of **Catalyst:**

The physical properties of pure and doped catalyst :Apparent porosity $(P_A\%)$, total pore Volume (V_P) , and density(D_P),at particle different temperatures (400,500,600,700,and 800) °C were determined by wet impregnation method (Satterfield, 1980): Weight of the catalyst grain dry (W1), and then immersed in boiling water for five

3-Determination the average particle size of the catalyst:

$d = K\lambda / \beta_{1/2} \cos \theta$

where d is the mean crystallite diameter, λ the X – ray wavelength(λ = 0.1545 nm), K the Scherrer constant (0.89), β 1/2 is the full – width at half maximum of the diffraction peaks of The particle size of the crystalline phases present in the different solids was calculated from the line broadening profile analysis of the main diffraction lines of these phases using Scherrer equation (Cullity, 1967): diffraction angle

spectrophotometer(AA-6300 supplied by shimadzu company).

5-Catalytic Activity of pure and Na₂O doped prepared Catalyst:

keep temperature of the reaction at a desired value.

The cell is fitted with ultraviolet radiation from a low pressure mercury lamp, type TQ150Z2 supplied by Karl Kolb Company. A schematic representation of the reaction system is shown in Figure (1), which was used in our previous works(Hussein,2007; Gassim,2007) The reaction results were studied by using UV-Visible Spectrophotometer(UV-1650 PC supplied by shimadzu company),at λ =541 nm. crystalline phases and θ is the **4-Analytical Study:**

The percentage of elements(Ni,Mn and Mg) in the prepared catalyst was determined by using atomic absorption

The catalytic activity of the prepared catalyst was tested, in the removal of the reactive red textile dye at different times(1,2,3, and 4 hrs.) at room temperature. In all experiments 50 mg of pure and Na₂O doped NiO-Mn₂O₃-MgO catalyst is suspended in 30 cm³ of the reactive red textile dye(50 ppm) ,which is placed in a photoreaction cell .The textile dye was kept homogeneous stirring with by magnetic stirrer. The cell contains side arms for passing air and for water circulation around the cell in order to



Figure (1): Schematic diagram for the photocatalytic reaction system

The X-ray diffractograms of pure NiO-Mn₂O₃-MgO prepared at 400 to 800 °C and single oxides were determined as shown in figures(2) and (3).and for Na₂O doped NiO-Mn₂O₃-MgO in figure(4).

Results and discussion 1-XRD Investigation of pure and Na₂O doped NiO-Mn₂O₃-MgO prepared at different temperatures:



Figure(3): XRD pattern for single NiO,Mn₂O₃,and MgO

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Figure(4): XRD pattern for Na₂O doped NiO-Mn₂O₃-MgO catalyst

calcination temperature in the range of 400-600°C led to а complete conversion of MnCO₃ into Mn₂O₃(Ghozza,2004), and the relative intensities of the diffraction lines of Mn₂O₃ increases and those of MgO decreases increasing by the temperature(El-Shobaky,2009), while The diffraction patterns of the sample prepared at 700 and 800°C become more broadening because of the sintering effect and overlapping of the oxides phases(Satterfield, 1976), The changes in the diffraction angle and relative intensities are summarized in table (1). These changes causes to the

From the comparison of X-ray diffraction pattern of the oxides in the catalyst and single show that there is a small deviation in the angles of diffraction and change in the relative intensity of the main absorption peaks(100%) these $are(2\theta)$ =32.99,33.01,32.95 and33.0 at 400,500,600,and 700°C respectively) ,but in 800°C become 36.11, this due to appear new phase of Mn₃O₄, and to the interaction between the mixed oxides in the catalyst .The best crystalline phases produced at 600°C and obtained a complete crystal size of the catalyst pattern . The rise in

decreases of the particle size of the catalyst as shown in figure(5) and table (2) that are calculated by using Scherrer equation (Cullity,1967): $d = K\lambda / \beta 1/2$ $\cos \theta$

Figure(5



):Average particle size(d) for NiO-Mn₂O₃-MgO Catalyst at different temperature(400 to 800) °C

the surface monolayer dispersion of Mn₂O₃, and Na₂O-doping enhanced the grain growth or particle adhesion of MgO, NiO and Mn₂O₃ crystallites to an extent proportional to the amount of dopant added (Leyrer et al.1996; Ghozza,2004; El-Shobaky et al,2002). The changes in the diffraction angle and relative intensities for NiO-Mn2O3-MgO doped catalyst are summarized table in

The figure(4) shows that the intensity of the diffraction lines and the particle size of Mn_2O_3 increased with the increasing of the amount of Na_2O added and increasing the degree of crystallinity of MgO phase. The effect of Na_2O is to lowers the thermal conversion process of Mn_2O_3 into Mn_3O_4 , and remained thermally stable even by heating at above 600 to 1000 °C, and leading to an effective decrease in the capacity of MgO, phase towards

(1).

Table(1): 2θ and Relative Intensities of Pure and Doped Catalyst at different temperature and different ratios of Na₂O

Pure Catalyst										
800°C		700°C		6	00°C	50	500°C		400°C	
20 for	20 in	20 for	20 in	20 for	20 in	1 20 for	20 in	20 for	20 in	
oxide in	standard	oxide in	standard	oxide	standard	l oxide	standard	oxide	standard	
the	oxide	the	oxide	in the	oxide	e in the	oxide	in the	oxide	
catalyst		catalyst		catalyst		catalyst		catalyst		
36.11	32.95	33.0	32.95	32.95	32.95	5 33.01	32.95	32.99	32.95	
Mn_3O_4	(100%)		(100%)		(100%))	(100%)		(100%)	
	Mn_2O_3		Mn_2O_3		Mn ₂ O ₂	3	Mn_2O_3		Mn_2O_3	
44.43	43.286	43.11	43.286	43.2	43.286	5 43.03	43.286		43.286	
(21%)	(100%)	(22%)	(100%)	(29 %)	(100%)) (11%)	(100%)		(100%)	
	NiO		NiO		NiC)	NiO		NiO	
44.43	42.9	43.18	42.9	43.2	42.9	9 43.03	42.9		42.9	
(21%)	(100%)	(22 %)	(100%)	(29 %)	(100%)) (11%)	(100%)		(100%)	
	MgO		MgO		MgC)	MgO		MgO	
			Ι	Doped (Catalyst					
8 %	Na ₂ O	6	% Na ₂ O		4 % N	Na ₂ O	2	% Na ₂	0	
20	2θ in	20 for o	xide	20 in	20 for	20 in	2θ for o	xide	20 in	
for oxide in	standard	in	the sta	ndard	oxide in	standard	in the cata	lyst	standard	
the catalyst	oxide	cata	alyst	oxide	the	oxide		•	oxide	
-			-		catalyst					
33.03,35.4	32.95	32.4,	35.0	32.95	33.16	32.95	31	2.45	32.95	
(100%,55%)	(100%)	(46%,10	0%) (100%)	(20%)	(100%)	(40%) Mı	n_2O_3	(100%)	
Mn ₂ O ₃	Mn_2O_3	M	n_2O_3	Mn_2O_3	Mn_2O_3	Mn_2O_3			Mn_2O_3	
43.08	43.286	4	3.14 4	43.286	43.30	43.286	43.	.201	43.286	
(15%)	(100%)	(2	(7%)	100%)	(41%)	(100%)	(4	0%)	(100%)	
NiO	NiO		NiO	NiO	NiO	NiO		NiO	NiO	
43.08	42.9	4	3.10	42.9	43.0	42.9	4	3.20	42.9	
(15%)	(100%)	(2	(7%)	100%)	(41%)	(100%)	(4	0%)	(100%)	

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| MgO |
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Temp.(°C)	$\beta_{1/2}$ (deg.)	deg.*[($\pi/180$)]*10 ⁴	d(A°)
400	0.350	609	234.7
500	0.300	522	273.8
600	0.250	435	328.6
700	0.275	478	298.7
800	0.450	783	184.1

Table(2): Average Particle Size for Cat.1 at different temperature.

2- Analytical Study:

The results of the percentage of elements(Ni,Mn,and Mg) ,and their oxides in the catalyst are shown in table (2).

Table(2): The results of the percentage of Ni,Mn,and Mg ,and their oxides in the catalyst

Sum. of percent. of mixed oxide in the catalyst %100	Percent of MgO	Percent. of Mg	Conc. of Mg (ppm)	Percent. of Mn ₂ O ₃	Percent of Mn	Conc. of Mn (ppm)	Percent of NiO	Percent of Ni	Conc. of Ni (ppm)	wt.of catalyst
99.92	39.40	23.612	41.00	50.03	17.41	1.511	10.49	8.245	0.7157	0.1085

volume(V_P),and particle density(D_P),respectively at different temperatures(400,500,600,700,and 800) °C ,that are calculated by using the data in tables (3) and (4):

2-Physical Properties of pure and doped Catalyst:

Figures(6),(7) and (8) show the physical properties of the pure catalyst :Apparent porosity (P_A %), total pore

Table(3): The physical properties of pure catalyst at different temperatures

Temp. °C	W1	W2	W3	Apparent Porosity (P _A %)	Pore Volume (V _P)	Bulk Density (D _p)
400	0.517	0.624	0.373	42.55	0.207	2.059
500	0.253	0.314	0.178	44.85	0.241	1.860
600	0.267	0.446	0.186	68.85	0.670	1.027
700	0.280	0.381	0.206	57.71	0.360	1.600
800	0.467	0.620	0.325	51.86	0.327	1.583

	inc physical	properties	of Doped	mici chi tempe	atures	
Ratio of	W1	W2	W3	Apparent	Pore Volume	Bulk Density
Na ₂ O in				Porosity (P _A	(V _P)	(D _p)
Catalyst				%)		-
2 %	0.301	0.373	0.189	39.13	0.239	1.636
4 %	0.272	0.378	0.151	46.69	0.389	1.198
6 %	0.277	0.407	0.146	49.80	0.469	1.060
8 %	0.293	0.436	0.159	51.62	0.488	1.057

Table(4): The physical properties of Doped catalyst at different temperatures







Figure(7):Pore Volume(cm³/g) of the catalyst at(400,500,600 700,and 800) °C

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Figure(8):Bulk Density(g /cm³) of the catalyst at(400,500, 600,700, and 800) °C

temperature above 600 to 800 C, the total pore volume and porosity, wil be decrease and the bulk density become more, because of sintering effect(Satterfield, 1976; Church and Joshi,1951; Thomas,1970).

The physical properties of doped catalyst will be increase with the increasing of

percentage of $Na_2O(2\%, 4\%, 6\%)$ and 8%) in the catalyst, as shown in figures (9),

When increasing the temperature from 400 to 600° C, increased the loss of carbon dioxide and water of crystallization and gets complete conversion of metal carbonate to metal oxide at a temperature of 600°C, and the size of the total pore volume and porosity will become more, therefore the weight of catalyst grain tablets decreased according to their volume ,this lead to decreasing of the bulk density. When increasing the



Figure(9): Apparent Porosity of the Na₂O doped catalyst

(10) and (11).

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Figure(10): Pore Volume(cm³/g) of the Na₂O doped catalyst



Figure(11): Bulk Density(g/ cm³) of the Na₂O doped catalyst 3- Catalytic Activity of pure and Na₂O doped NiO-Mn₂O₃-MgO catalyst

The catalytic activity of the pure and doped catalyst was tested in the photodegradation of textile reactive red dye at different time(1,2,3 and 4 hrs.) as shown in figures (12) and (13) respectively.



Figure(12): UV-visible Spectrum for the Reactive Red Dye at room temperature by using doped NiO-Mn₂O₃- MgO catalyst in different times :a-pure dye b-dye after adsorption c-(1hr.) d-(2hr.) e-(3hr.) f-(4hr.)



The results are also showed that the catalytic activity of Na₂O doped catalyst is more than that for pure catalyst, with respect to the time that is needing to complete degradation of dye this may be attributed to the increasing of surface area of this dopant catalyst and to the decreasing of activation energy (El-Shobaky et which equal to (13 kJmol⁻ al.,1997), ¹) compared with that for the pure catalyst(17.8 kJmol⁻¹) as shown in figure (15). The observed increase in the catalytic activity of the employed system due to Na₂O-doping conducted at 600°C could be attributed to creation of new ion pairs(Na⁺ -Mn³⁺)acting as active sites for the dye decomposition. be attributed .and also to the dissolution of very small amount of lattice of Na₂O in Mn_2O_3 ,and $Na^+(Mn^{3+})$ represent location of sodium ion in the position of host cation lattice ions ofMn₂O₃(Kröger,1964)

Figure(13): UV-visible Spectrum for the Reactive Red Dye at room temperature by using doped NiO- Mn_2O_3 -MgO catalyst b-dye after adsorption in different times :a-pure dye c-(15min.) d-(30min.) e-(45min.) f-(60min) (105 min.) k-(120 min) g-(75min.) h-(90min.) i-

From the Experimental results of UV-Visb.analysis that is showing in figures(12) and (13) indicated that the decolorization of dyes is facilitated in the presence of catalyst and increase with increasing of time. The rate of decolorization was recorded with respect to the change in the intensity of absorption peak in visible region. The prominent peak was observed at λ_{max} , 541 nm which decreased gradually and finally disappeared indicating that the dye had been degraded.



Figure(15):activation energy for pure and Na₂O doped catalyst

leaving positive holes in the valence band, and forms the electron-hole pair on the surface of catalyst(eq.1).The high oxidative potential of the hole(h^+_{VB}) in the catalyst permits the direct oxidation of the dye to reactive intermediates(eq.2).

$$h^+_{VB} + dye \rightarrow dye^{*+} \rightarrow oxidation of dye$$
(2)

Another reactive intermediate which is responsible for the degradation is hydroxyl radical (OH^{*}). It is either formed by the decomposition of water (eq.3) or by reaction of the hole with

increase calcination The in temperature within 400°C and 700°C and doping with different amounts of Na₂O did not change the mechanism of the catalyzed reaction ,and did not change the dissipation of active sites, character i.e. the of surface heterogeneity. In other words, these treatments did not change the energetic nature of active site but increased the concentration of active sites participated in the catalyzed reaction Ghozza,2004; El-Shobaky et al,2009)

The mechanism of photocatalyzed decolorization of a dye in solution include the photoexitation of the semiconductor ,the electrons in the semiconductor are exited from the

valence band to the conduction band

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 OH^- (eq.4). The hydroxyl radical oxidize the dye molecules, which leads to the partial or complete degradation of the dye(eq.5) (Daneshvar,2003).

 $h^+_{VB} + H_2O \rightarrow H^+ + OH$

.....(3)

Conclusions :

 The best crystalline phases of the prepared catalyst produced at 600°C and obtained a complete crystalanity of the catalyst pattern and the best physical properties .
The results are also showed that the

catalyst is more than that for pure catalyst.

3-The activation energy of doped catalyst is lower than that for pure catalyst

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تحضير ودراسة العامل المساعد NiO-Mn₂O₃-MgO المجرد والمشوب باوكسيد الصوديوم

Na2O نجلاء ناصر حسين صالح هادي كاظم قسم الكيمياء –كلية العلوم- جامعة بابل

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تاريخ الاستلام:- 2012/10/30

الخلاصة:

حضر العامل المساعد MgO-MgO-MgO، بالنسبة 10:50:40 لمزيج الأكاسيد على التوالي باستخدام طريقة الترسيب المشترك لمزيج كاربونات الفلزات الثلاثة من محلول نتراتها بإضافة 1 مولاري من محلول بيكاربونات الصوديوم عند دالة حامضية ثابتة .تم تجفيف الكاربونات عند 101 م⁰ وتم تحميصها عند درجات حرارة مختلفة (800,700,600,500,400) م⁰ لمدة 4 ساعات .تم تحضير مزيج الأكاسيد المشوب باوكسيد حرارة مختلفة (800,700,600,500,400) م⁰ لمدة 4 ساعات .تم تحضير مزيج الأكاسيد المشوب باوكسيد كميات مختلفة (800,700,600,500,400) م⁰ لمدة 4 ساعات .تم تحضير مزيج الأكاسيد المشوب باوكسيد كميات مختلفة من نترات الصوديوم (800,700,600) م⁰ لمدة 4 ساعات .تم تحضير مزيج الأكاسيد المشوب باوكسيد كميات مختلفة من نترات الصوديوم (800,700,600) م⁰ لمدة 4 ساعات .تم تحضير مزيج الأكاسيد المشوب باوكسيد ورارة مختلفة من نترات الصوديوم (800,700,600) والمنغنيز والمغنينز والمغنيسيوم 2(600) م⁰ وتم وتم والصوديوم بتشبيع وزن مجفف من مزيج بيكاربونات النيكل والمنغنيز والمغنيسيوم 2(60,600) م⁰ وتم والموديوم بتشبيع وزن مجفف من مزيج بيكاربونات الايكان والمنغنيز والمغنيسيوم 2(60,600) م⁰ وتم وربع مديات النيكل والمنغيز والمعنيسيوم 2(60,600) م⁰ وتم والموديوم (800,700,600) وربعالية الامتصاص الذري، طيف الأشعة فوق البنفسجية تحميصها في درجة 600 م⁰ لمدة 4 ساعات. درست خواص السطح والخواص الدلوي، طيف الأشعة فوق البنفسجية والمرئية وطريقة التشبيع الرطب .تزداد درجة تبلور الطور الناتج وحجمه البلوري كدالة لزيادة درجات الحرارة من والمرئية وطريقة التشبيع الرطب .تزداد درجة تبلور الطور الناتج وحجمه البلوري كدالة لزيادة درجات الحرارة من مام والمرئية وطريقة التربيع الرطب .تزداد درجة تبلور الطور الناتج وحجمه البلوري كالة لزيادة درجات الحرارة من مام والمرئية فوق البنفسجية ، ولمرئية فوق البنفسجية ،ودراسة تأثير درجات المرازة على 600 م⁰ ، ولكنها نقل عند درجتي الحرارة 700 و 800 م⁰ .تم ولاسة تأثير درجات المساعد المجرد والمشوب في تكمير الصبغة الفعالة الحمارء بوجود الأشعة فوق البنفسجية ،ودراسة تأثير درجات المرازة على فعالية النفار المامل المساعد الممود والمشوب في تكمير الصبغة الفعاليا المساعد المجرد والمقوب في تكير الصبغة الفعال المام المساعد المجرد طاقة تشيط تساوي ¹ المامل المساعد المجرد طاقة ت