Hungarian Society of Textile Technology and Science
Textilipari Műszaki és Tudományos Egyesület (TMTE)

34th Hungarian Textile Conference

Budapest, 4-6 June 1991.

TEXTILIPARI MŰSZAKI ÉS TUDOMÁNYOS EGYESÜLET

HUNGARIAN SOCIETY OF TEXTILE TECHNOLOGY AND SCIENCE
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SOCIÉTÉ HONGROISE DE TECHNOLOGIES ET SCIENCES TEXTILES
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Title of lecture / Die Adresse des Vortrages:
.Optimization.of.Hercosett/Optical.Brightener/ Hydrogen.Peroxide.Systems.Applied.to.Untreated.Wool.For.Shrinkproofing.

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1st CIRCULAR

34th Hungarian Textile Confcrence
4–6. June 1991. Budapest, Hungary

The Hungarian Society of Textile Technology and Science organizes on **4–6. June 1991** in Budapest

THE 34th HUNGARIAN TEXTILE CONFERENCE.

The main subject of the Conference is:
"THE HUNGARIAN TEXTILE INDUSTRY AFTER 1992."
With this title we intended to point at the marking change which will take place in the European textile industry as a consequence of the integration of the European Community.
Within the frame of the main subject we intend to discuss following areas:

- the change which may be expected in the raw material structure
- the change which may be expected in the product structure
- new processing processes and technologies /in all phases of the textile industry/
- new finishing and make-up methods
- methods to increase the productivily
- requirements towards product quality
- protection of consumers' interests
- training of experts for the textile industry
- preliminary information on ITMA 91.

The Organizing Committee experts about 4-500 participants to the Confe-rence, from the representatives of the domestic and international textile industry.

At the Confercrice 60-70 lectures arc expected to be giveri in 3 sections. Official languages of the Conference: **Hungarian, English, German.** Simul-taneous translation will be provided for each language.

In case if you intend to give a lecture at the Conference on one of the subjects listed above, so please send us the title and a brief synopsis of your lecture on the enclossed Registration Form till **30. November 1990** to the Organizing Committee. Address: H-1027 Budapest, Fő u. 68.

Best wishes

Lakatos Gy.

Katalin Lakatos Győri
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Report n° 7

OPTIMIZATION OF HERCOSETT/ÒPTICAL BRIGHTENER AGENTS/HYDROGEN PEROXIDE SYSTEMS APPLICATIONS TO UNTREATED WOOL FOR SHRINKPROOFING

APPIED

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ABSTRACT

We have studied the application on untreated wool fibers of different aqueous dispersion systems made with a cationic resin Hercosett , three commercial anionic optical brightener agents (O.B.A.), (two estylbensulphonic acid derivatives, Uvitex CF-200 and Blankophor BA liq. B and a dyestearylbiphenyl derivative, üvitex NFW) and the oxidative agent hydrogen peroxide, in order to impmve both whiteness and shrinkproofing properties of wool fibers with a lesser damage of keratinic structure. Time and temperature of treatments have been optimized using the central composite rotatable desing of Box and Hunter using in all cases a weight ratio of Hercosett/O.B.A. that produced a higher shrinkproofing pmperties on wool samples. Results obtained for each O.B.A. investigated have been also compared in order to determine the optimum application.

INTRODUCTION

Many chemical compounds have been described in the literature as fluorescents and in the last decades intensive research has yielded many more fluorescent compounds which are suitable for their whitening effect (1,919 Optical brightener agents (O.B.A.) absorb the ultraviolet portion of the daylight spectm invisible to the eye, and convert the energy thus taken up into the longer-wavelength visible portion of the spectm, i.e., into blue-violet light.

Hercosett resin is a reactive cationic crosslinked polimer (10,11), capable of

forming insoluble aggregates in aqueous media in the presence of anionic surfactants, provided the amount of surfactant is maintained between certain limits (5,6). Previously we investigated the use of anionic surfactants in applying Hercosett resin to untreated wool fabrics to impart shrink-proofing properties (7).

A mixture of Hercosett resin and different anionic optical brightener agents (O.B.A.) in an aqueous medium may form insoluble aggregates depending of the ratio of the two species (14). Insoluble aggregates are formed by ionic interaction between the two components, and when the brightener concentration is increased, the aggregates become soluble. Hydrophobic linkages are then also implicated (3).

In an earlier paper, we described a process to minimize the shrinkage of wool fibers, based on the formation of a complex of Hercosett resin, the anionic optical brightener agent Uvitex NFW and hydrogen peroxide as oxidative agent, and its interaction with untreated wool fiber. In this way, in addition to providing wool fibers with satisfactory shrinkproofing properties, their brightness characteristics could be substantially improved. Likewise, we studied the degradative effects on the fibers caused by applying these systems on knitted wool fabrics and optimized the results obtained (4).

In the present work, we have compared the results of the application on untreated wool fibers of different dispersion systems formed by Hercosett resin, hydrogen peroxide and three commercial anionic optical brightener agents (O.B,A.), two estylbensulphonic acid derivatives, Uvitex CF-200 and Blankophor BA Liq. B, and a dyestearylbiphenyl derivative, Uvitex NFW. Time and temperature parameters have been optimized for each O.B.A. in the range of time and temperatures between 5 and 20 minutes and 35ºC and 80 ºC using in all cases a weight ratio of both O.B.A. and resin components that promotes a higher shrinkproofing properties on treated wool samples.

EXPERIMENTAL

Materials

Wool sample was a fabric knitted to a cover factor of 1.2 from R72-tex/2

botany wool yarns. The resin Hercosett 57, which is a polyamide-epichlorhydrin resin in a 10% active aqueous solution was manufactured by Hercules Chemical Co. Optical brightener agents (O.B.A.) were Blankophor BA Liq.B (estylbendisulphonic acid derivative), C.I. Fluorescent Brightener 113, manufactured by Bayer S.A., Uvitex CF-200 (estylbensulphonic acid derivative), C.I. Fluorescent Brightener 134, and Uvitex NFW (dyestearylbiphenyl derivative) from Ciba-Geigy S.A. The oxidative agent (hydrogen peroxide) used in the system was a 30% (w/v) aqueous solution (Probus S.A.)

Testing methods

Shrinkage of wool fabrics was tested at a liquor ratio of 30:1 and a 1 Kg load for 3 hours at 40ºC; the pH was held at 7.5 using a phosphate buffer according to the superwash test (15).
The cysteic acid and the alkaline solubility of the treated wool fabrics were determined in accordance with the methods of the International Wool Textile Organization (17,18). The ball penetration resistance loss (expressed as BPR in %) of treated samples was evaluated with a pendulum instrument (13). The whiteness index of the treated samples was estimated by Elrepho photometer, using the tristimulus colorimetry technique (8).

Treatments with chemical agents

Dispersion systems consisting of Hercosett resin/O.B.A./Hydrogen peroxide were prepared by progressively adding of increasing amounts of each O.B.A. aqueous solution to the solution containing Hercosett resin (2g/l) and 2 volume concentration of hydrogen peroxide with vigorous stirring at 20ºC in a glass container, at pH value adjusted to 7.0.
Untreated wool samples (10g) were then inmersed in these aqueous ternary dispersion systems and treated for 20 minutes at 20ºC with occasional stirring, bath ratio 1:30, and pH 7.0. After treatments, the samples were extracted to approximately 80% pickup and then dried and cured in a forced air oven at 80ºC for 60 minutes.

RESULTS AND DISCUSSION

Treatments of wool fabrics with O.B.A./Hercosett/Hydrogen peroxide systems

Figure 1 shows the shrinkage area percentage of the knitted wool samples treated with a ternary systems freshly prepared made with Hercosett resin (2g/l), hydrogen peroxide (2 volume), and increasing amounts of Uvitex NFW, Uvitex CF-200 and Blankophor BA Liq. B, (ranging from 0.1 to 6 g of O.B.A. per g of Hercosett resin). Amounts of the O.B.A. from 1.5 to 3.5 g per g of Hercosett resin enhances shrinkage properties in untreated wool, attaining values lesser than 10%, which is the maximum acceptable in accordance with the super wash test of the Wool Foundation (15). Exceeding amounts of O.B.A. of 4 g per g of Hercosett resin, produce dispersions with smaller optical density values, which do not enhance shrinkproofing properties when applied to untreated wool, specially for Hercosett/Uvitex NFW system.

Statistical treatments of the experimental results

Since the hydrogen peroxide reagent exerts an oxidizing influence on the keratinic structure of wool fibers, we studied some physicochemical parameters of treated wool that provide a suitable measure of its oxidation level, including shrinkproofing values, whiteness index, ball penetration resistance loss, cysteic acid content, and alkaline solubility of treated samples. These studies were carried out using the central composite rotatable design of Box and Hunter (2) for two variables , time (X_1) and temperature (X_2), the experimental levels of which are shown in Table I. The following parameters were constant: Hercosett resin concentration (2g/l), hydrogen peroxide concentration (2 volume) O.B.A. concentration (2 g/ g Hercosett resin), bath ratio (1:30). The results obtained are shown in Table II, and in Table III the regression coefficients in codified variables are given, as well as the significance of the regression coefficients, and the lack of fit obtained in the variance analysis.

Bearing in mind that the lack of fit is not significative in any of the parameters given in Table III, the response surfaces satisfactorily represents

the studied properties in function of treatment time and temperature. Besides the first degree terms and/or second degree terms of the adjusted equations are significative at 1%, 5% or 10% level, except for the % Shrinkage Area in presence of Blankophor. However, taking into account that both F-Shedecor value obtained in the regression analysis of variance test (ANOVA) (12) for the 1st and 2nd degree terms have a significative level near to a 10% we can state that also in this case the adjusted surface adequately represents the phenomenon. Larger dispersion observed in the five central replicates could explain this lower significative level for the % Shrinkage Area for Blankophor-Hercosett system.

To obtain the experimental conditions which lead to an acceptable level of both shrinkproofing properties and whiteness degree with a lesser physicochemical damage of treated wool fibers, we represent graphically the surface response for each O.B.A./Hercosett system in a range of time and temperature that includes the shrinkage acceptable level (lower than 10%) for the three O.B.A./Hercosett systems considered, that is to say time values from 5 to 20 minutes and temperature values between 35ºC and 80ºC.

Figure 2 shows these optimal conditions for Blankophor BA liq. B/Hercosett system that in a short time and low temperature is obtained, and for Uvitex CF-200/Hercosett and Univex NFW/Hercosett systems that a wider optimal zone (except in a short time range or a long time and low temperature range) are obtained.

During applications of Hercosett/H_2O_2/O.B.A. complexes on untreated wool knitted samples two phenomena take place simultaneously. First, the attraction and fixation of O.B.A./Hercosett complexes on wool, which promotes an improvement of wool physical properties, and second, an oxidative effect due the presence of the oxidising agent hydrogen peroxide that promotes the formation of active sites of cysteic acid in wool where O.B.A./Hercosett complexes could be ionically linked which also could affect the stability of O.B.A./Hercosett disperse systems. As a consequence, the following discussion will be presented following the results stated in Figures 3-7, in an attempt to establish any relation between the application of O.B.A./Hercosett dispersed systems and any especific improvement resulting of the two phenomena previously indicated when Hercosett/H_2O_2/O.B.A. complexes are applied on

untreated wool.

Shrinkproofing properties

Figure 3 shows that in short times range the influence of the temperature on the % shrinkage area are the same whichever O.B.A./Hercosett system considered obtaining the best results with Blankophor BA liq. B/Hercosett system. For longer treatments percentage of shrinkage area increases in Blankophor BA liq. B/Hercosett system, whereas with the two other Uvitex/Hercosett systems decreases in a similar way reaching an optimal stable zone. For longer treatments in Blankophor BA liq. B/Hercosett system shrinkage increases with temperature; being the influence of the temperature on shrinkage percentage stronger as time increases.

For the two Uvitex/Hercosett systems the effect of temperature on percentage of shrinkage area decreases when time increases reaching a slight reverse effect. In this case higher temperatures leads to a better shrinkproofing properties.

The acceptable shrinkage zone (lower than 10%) in Blankophor BA liq. B/Hercosett system would be bounded for time values between 5 and 11 minutes and temperature values between 37ºC and 62ºC, whereas in Uvitex NFW/Hercosett system would be for treatments longer than 8-10 minutes whichever temperature was, although for short time range temperature has to be lower than 70ºC and for long time range temperature has to be higher than 50ºC.

As it has be seen, we can establish two clear contradictory tendencies. Blankophor BA liq.B/Hercosett systems promotes an improvement in wool shrinkproofing properties at low time and temperatures whereas the application of Uvitex NFW/Hercosett and Uvitex CF-200/Hercosett promotes shrinkproofing properties at higher time and temperature conditions (Figure 3). These results could be explained considering both the physico-chemical stability of these systems with regard to the oxidative action of the hydrogen peroxide reagent in the range of temperatures and times studied and the influence of these parameters in the attraction and fixation of those dispersed systems on wool.

Ball penetration resistance loss

Considering the ball penetration resistance loss (BPR%) of samples treated with O.B.A./Hercosett systems, we can see that BPR% increases with time and temperature (Fig. 4) in a similar way that cysteic acid content (Fig. 6) and alkaline solubility (Fig. 7) does, being 10% the maximun value accepted in the ball penetration resistance loss test (13).

It can be observed that for Blankophor BA liq. B/Hercosett system, values of BPR higher than 10% were obtained in the range of time and temperature where any shrinkproofing effect was obtained. Nevertheless with both Uvitex/Hercosett systems unacceptable BPR% values higher than 10% were obtained with treatments longer than 15 min and higher than 65ºC.

Probably, improvements in BPR% and shrinkproofing properties among other factors depends on the surface deposition and fixation of the O.B.A./Hercosett complexes on wool fibre surfaces which could improve surface resistence and consequently mechanical properties.

Whiteness degree

Figure 5 shows the white level indicated as whiteness index (%) being the 100% level the degree of whiteness obtained for samples treated with O.B.A. using treatment conditions recommended by the manufacturer.

Refering to the whiteness index values, a similar improvement was obtained in applications for each dispersion systems as a function of time and temperatures. This effect could be imputed both to the logical exhaustion on wool of the free O.B.A. molecules in equilibrium with those complexed with the Hercosett resin and the bleaching effect due to the oxidative action of the hydrogen peroxide during the treatments on wool fibres (Figure 5)

As Figure 5 shows time has lower influence on whiteness index than temperature. With temperatures higher than 70ºC whiteness index higher than 100% could be obtained.

Chemical parameters

In order to detect the chemical degradative effect of the presence of hydrogen peroxide in the system, besides the ball penetration resistence loss above mentioned, the cysteic acid content and the alkaline solubility of the treated wool samples obtained were studied.

Both the the cysteic acid content (Fig. 6) and the alkaline solubility (Fig. 7) are influenced by treatment time and temperature, increasing with time and temperature in a linear way. These two parameters show an acceptable level of fiber chemical modification. In no case did the cysteic acid content exceed 1.5% nor the alkaline solubility 28% (16,20,21).

Considering these chemical parameters a similar tendency in the oxidative damage in all cases independently of the O.B.A. agent used is observed.

CONCLUSIONS

From the results reported in this paper, we can conclude that the application of Hercosett resin on untreated wool knitted fabrics via Hercosett/anionic optical brightener agent systems including an oxidising reagents as hydrogen peroxide promotes an improvement in both shrinkproofing and whiteness properties applying these systems within a certain margin of weigth relative ratio of both resin and O.B.A. components with a minimun deterioration in the keratin structure.

The application on untreated wool fibers of complexes formed by Hercosett resin, hydrogen peroxide and three different O.B.A., two estylbensulphonic acid derivatives (Blankophor BA liq B and Uvitex CF-200) and a dyestearylbiphenyl derivative (Uvitex NFW) has been optimized being the range of treatment time and temperature values between 5 and 20 minutes and 35ºC and 80ºC, respectively. Treatments promoted in all cases an improvement both in shrinkproofing and whiteness properties. Hercosett/Blankophor BA liq B complexes was the most unstable system in front of the oxidising reagent, improving shrinkage and whiteness of treated samples only at low time and temperature treatment conditions, whereas both Hercosett/Uvitex systems were more stable promoting shrinkage and whiteness improvement at higher time and

temperatures.

The physicochemical damage of treated wool fibers was in all cases directly dependent of both optimized parameters time and temperature being only slightly dependent of the O.B.A. used in each system investigated.

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FIGURE LEGENDS

FIGURE 1. Shrinkage area percentage of wool samples treated with O.B.A./Hercosett/H_2O_2 complexes versus O.B.A./Hercosett ratio.

FIGURE 2. Box Hunter optimization response surfaces diagram of shrinkage area percentage (10%) of wool samples treated with O.B.A./Hercosett/H_2O_2 versus treatment time and temperature.

FIGURE 3. Spatial response surfaces of shrinkage area %, of treated wool fabrics with different O.B.A./Hercosett/H_2O_2 complexes versus time and temperature.

FIGURE 4 Spatial response surfaces of ball penetration resistance loss of treated wool fabrics with different O.B.A./Hercosett/H_2O_2 complexes versus time and temperature.

FIGURE 5. Spatial response surfaces of whiteness index of treated wool fabrics with different O.B.A./Hercosett/H_2O_2 complexes versus time and temperature.

FIGURE 6. Spatial response surfaces of cysteic acid of treated wool fabrics with different O.B.A./Hercosett/H_2O_2 complexes versus time and temperature.

FIGURE 7. Spatial response surfaces of alkalyne solubility of treated wool fabrics with different O.B.A./Hercosett/H_2O_2 complexes versus time and temperature.

TABLE II. Results of the parameters studied in the central composite rotatable design of Box and Hunter for the variables of time (X_1) an temperature (X_2) in the application of Hercosett/O.B.A./hydrogen peroxide aqueous systems on untreated knitted wool samples.

Treatment	Values of variables		% BPR			% Alkaline solubility			% Shrinkage Area			% Whiteness index			% Cysteic acid		
	X_1	X_2	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
1	-1	-1	1.0	1.7	1.8	18.2	19.0	18.5	15.4	12.8	15.0	46	47.7	48	0.57	0.59	0.54
2	1	-1	4.6	5.7	7.0	19.8	20.5	20.0	16.-	12.0	14.0	46	45.6	46	0.78	0.80	0.80
3	-1	1	4.6	5.7	7.0	19.5	20.4	19.5	19.2	15.6	14.8	98	99.8	97	0.81	0.84	0.80
4	1	1	10.0	9.7	12.0	24.8	25.4	24.5	9.5	7.6	17.0	110	108.0	108	1.29	1.32	1.35
5	-1.414	0	2.6	2.7	3.3	16.2	17.2	16.2	17.8	15.6	8.0	70	68.0	67	0.80	0.80	0.85
6	1.414	0	8.0	8.5	10.8	22.0	22.0	21.5	11.5	9.2	17.0	75	73.0	75	0.91	0.96	0.97
7	0	-1.414	2.6	2.8	3.3	19.7	20.1	19.5	14.5	12.0	10.0	40	38.0	39	0.54	0.59	0.54
8	0	1.414	8.0	8.5	10.5	24.0	24.0	23.5	12.9	10.8	14.0	120	118.0	118	1.10	1.17	1.19
9	0	0	3.8	3.6	5.1	18.0	19.0	18.0	10.0	8.0	13.0	55	50.0	54	0.75	0.76	0.87
10	0	0	3.6	3.8	5.2	21.0	20.0	21.0	8.0	9.0	8.0	58	53.0	55	0.95	0.97	0.69
11	0	0	3.7	3.4	4.9	18.0	18.0	19.0	9.0	7.0	10.0	53	51.0	53	0.84	0.87	0.98
12	0	0	3.2	3.6	4.8	19.0	21.0	17.0	9.0	8.0	8.0	54	52.0	56	0.79	0.79	0.80
13	0	0	3.9	3.7	5.2	20.0	19.0	19.0	8.0	9.0	9.0	55	51.0	54	0.90	0.88	0.80

A - Uvitex CF200

B - Uvitex NFW

C - Blankophor BA

TABLE III. The regression coefficients in codified variables, significance level of the regression coefficients and the lack of fit deviation of the adjustment obtained in the variance analysis.

Parameter	O.B.A.	Independent terms	Regression coefficients					Significance level		
			X_1	X_2	X_1^2	X_2^2	X_1X_2	1st degree terms	2nd degree terms	lack of fit
% BPR	A	3.61	2.063	2.045	0.995	1.020	-0.075	1%	1%	NS
	B	3.64	2.079	2.079	0.768	0.768	0.450	1%	1%	NS
	C	5.04	2.601	2.548	0.999	0.924	-0.050	1%	1%	NS
% Alkaline	A	19.20	1.888	1.548	-0.025	1.350	0.925	5%	NS	NS
Solubility	B	19.40	1.661	1.477	0.225	1.450	0.875	5%	NS	NS
	C	18.80	1.749	1.395	0.138	1.463	0.875	10%	NS	NS
% Shnrinkage	A	8.80	-2.251	-0.620	3.138	2.663	-2.575	1%	1%	NS
Area	B	8.20	-2.231	-0.412	2.125	1.625	-1.800	5%	5%	NS
	C	9.60	1.941	0.857	2.188	1.938	0.800	NS	NS	NS
% Whiteness	A	55.00	2.384	28.640	8.438	12.188	3.000	1%	1%	NS
index	B	51.40	1.646	28.453	9.806	13.556	2.575	1%	1%	NS
	C	54.40	2.539	27.838	8.300	12.050	3.250	1%	1%	NS
% Cysteic	A	0.846	0.106	0.193	0.011	-0.007	0.068	5%	NS	NS
acid	B	0.854	0.115	0.199	0.015	0.015	0.068	1%	NS	NS
	C	0.818	0.122	0.216	0.042	0.020	0.073	5%	NS	NS

A: Uvitex CF200

B: Uvitex NFN

C: Blankophor BA

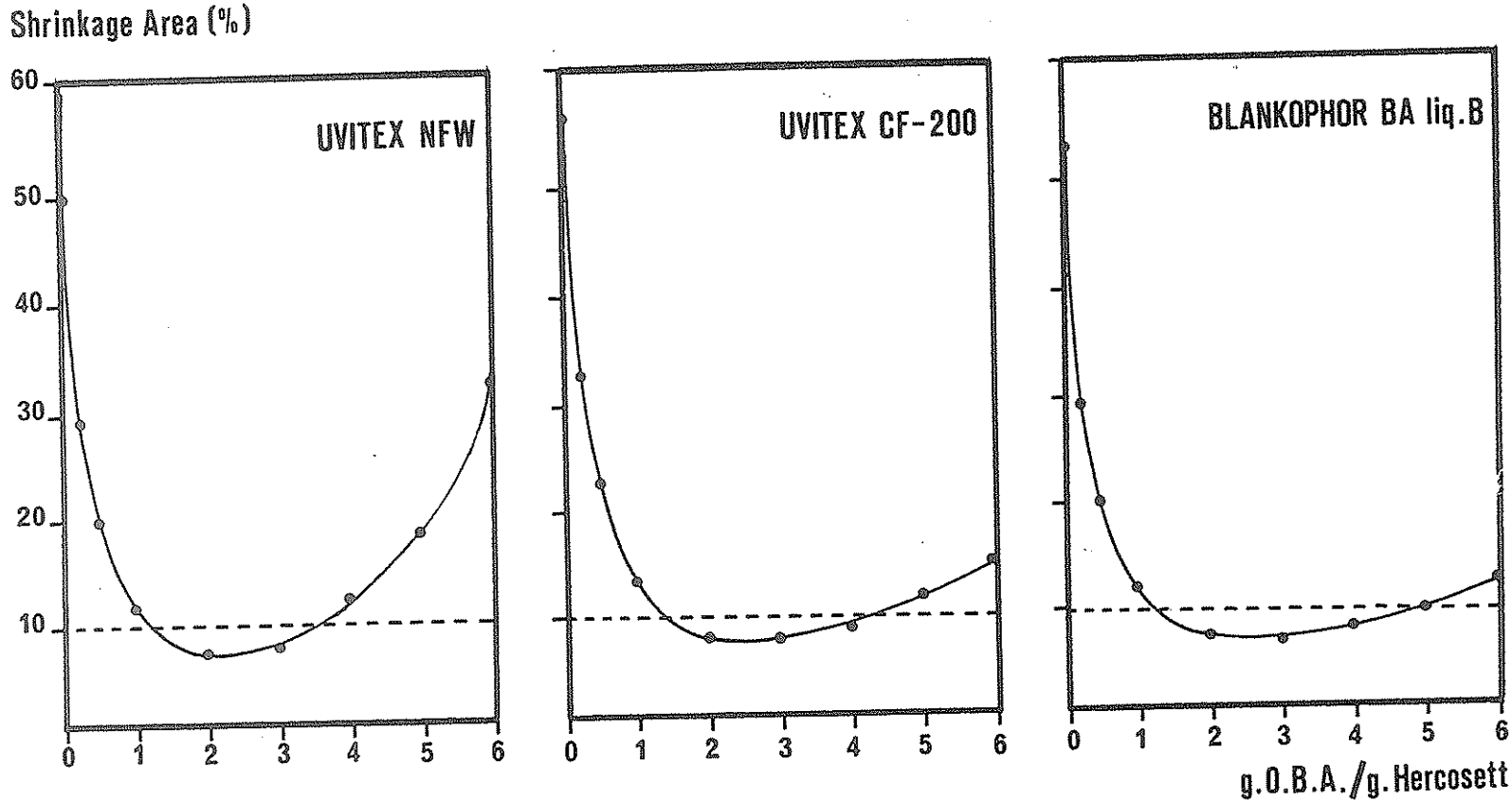
Shrinkage Area (%)
60
50
40
30
20
10
UVITEX NFW
UVITEX CF-200
BLANKOPHOR BA liq.B
0 1 2 3 4 5 6
g.O.B.A./g.Hercosett

FIGURE 1

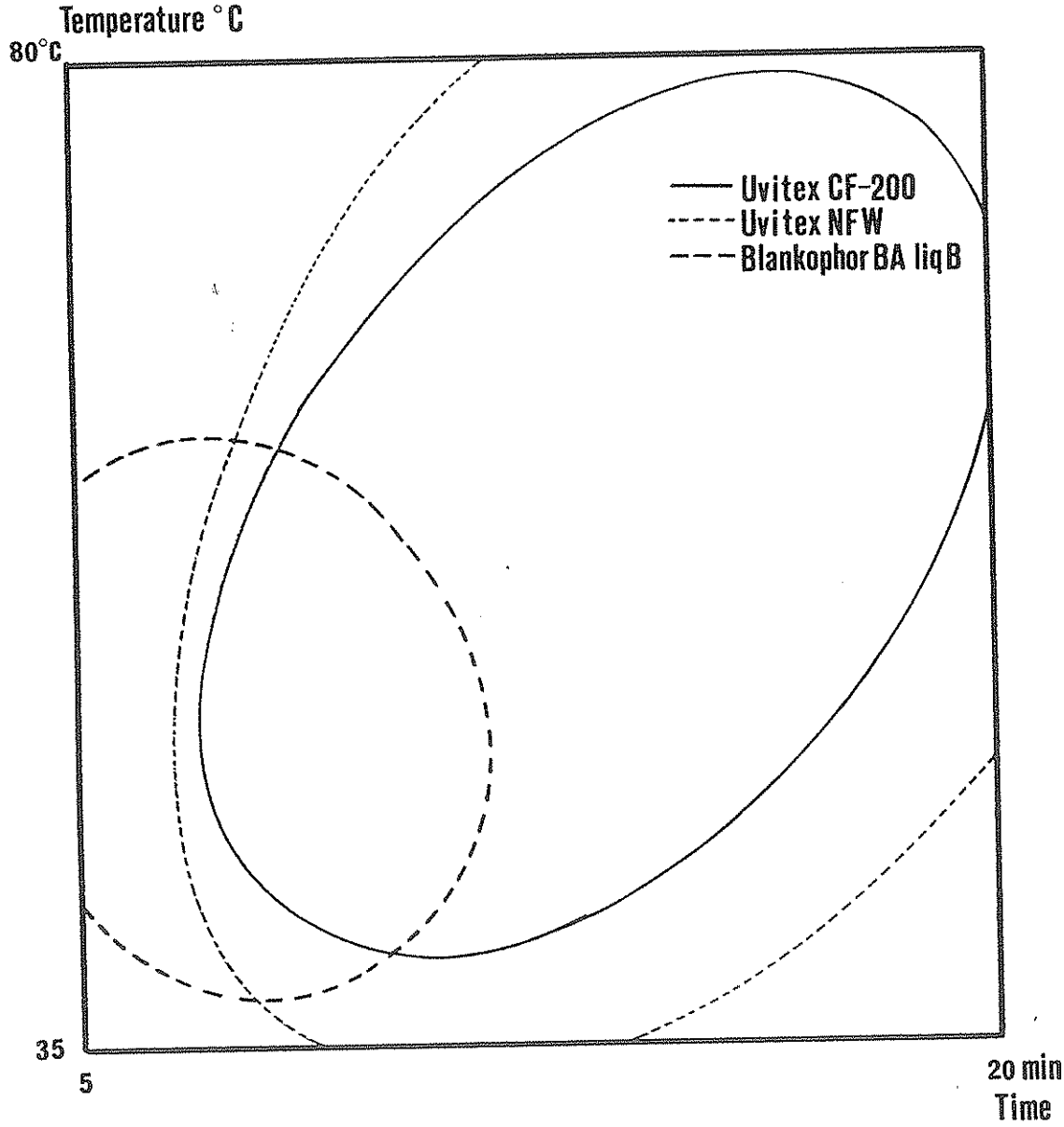
Temperature °C
80°C
Uvitex CF-200
Uvitex NFW
Blankophor BA liq B
35
5
20 min
Time

FIGURE 2

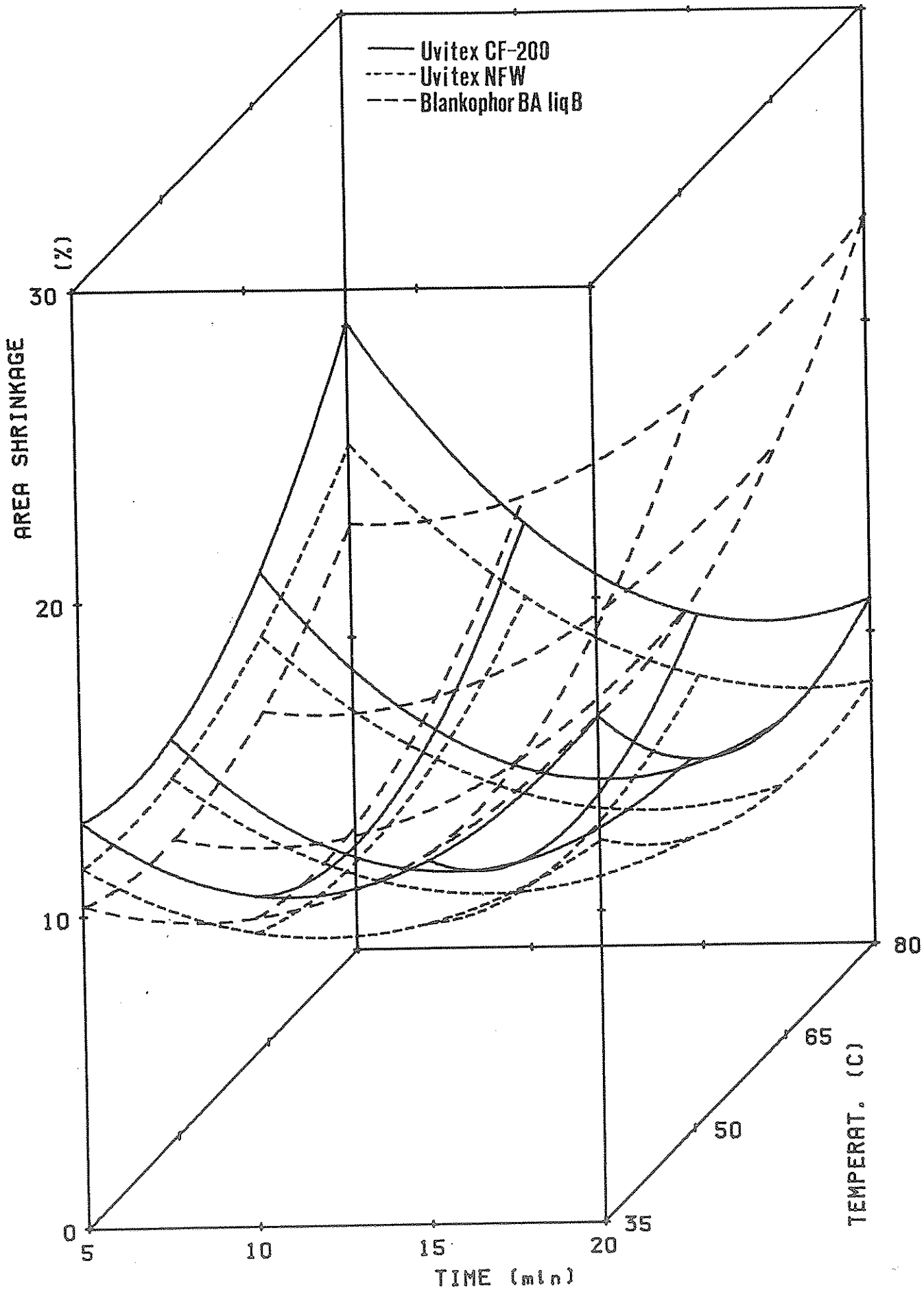
Uvitex CF-200
Uvitex NFW
Blankophor BA liq B
AREA SHRINKAGE (%)
30
20
10
0
5
10
15
20
TIME (min)
35
50
65
80
TEMPERAT. (C)

FIGURE 3

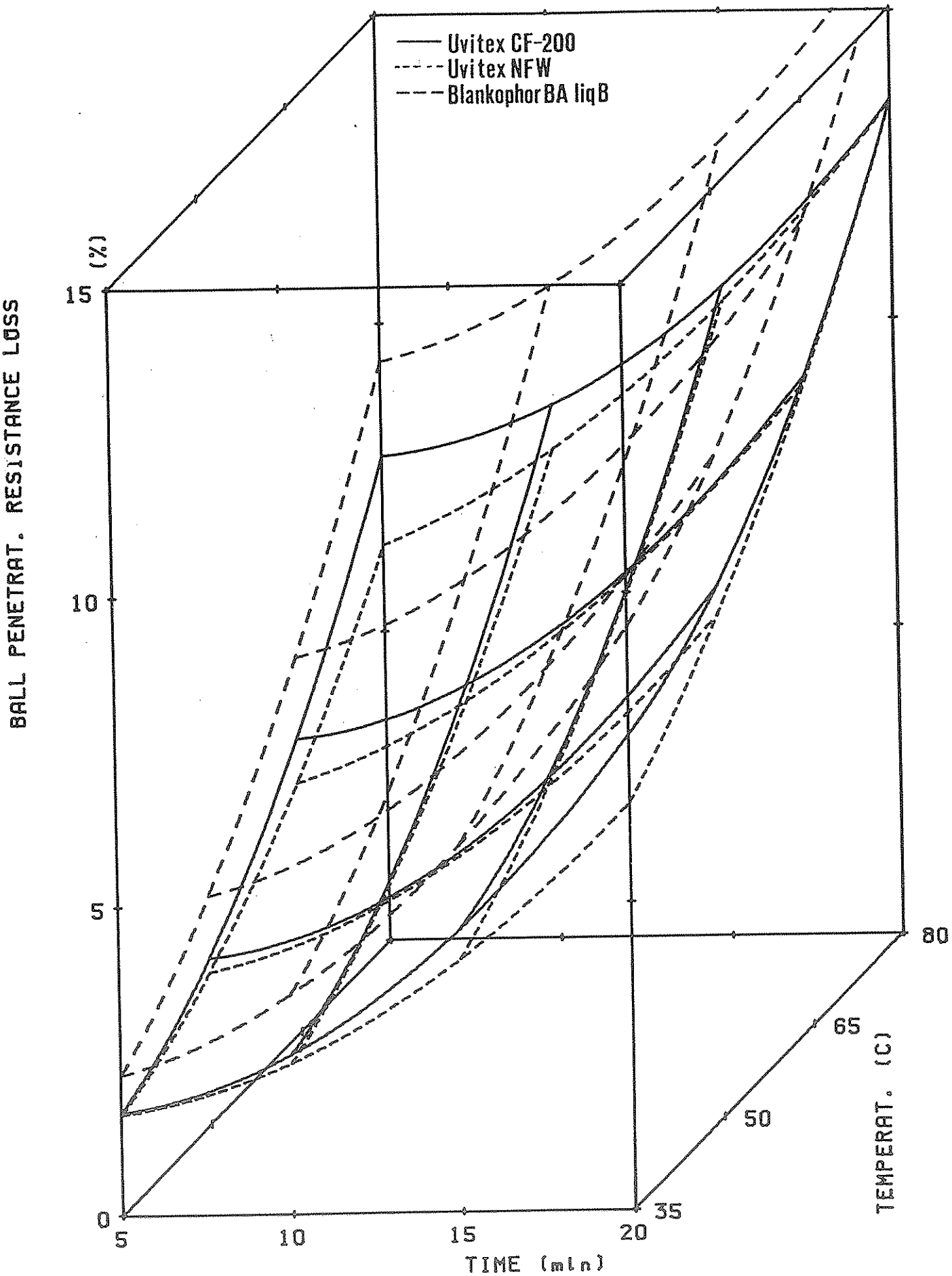
Uvitex CF-200
Uvitex NFW
Blankophor BA liq B
BALL PENETRAT. RESISTANCE LOSS (%)
15
10
5
0
5
10
15
20
TIME (min)
35
50
65
80
TEMPERAT. (C)

FIGURE 4

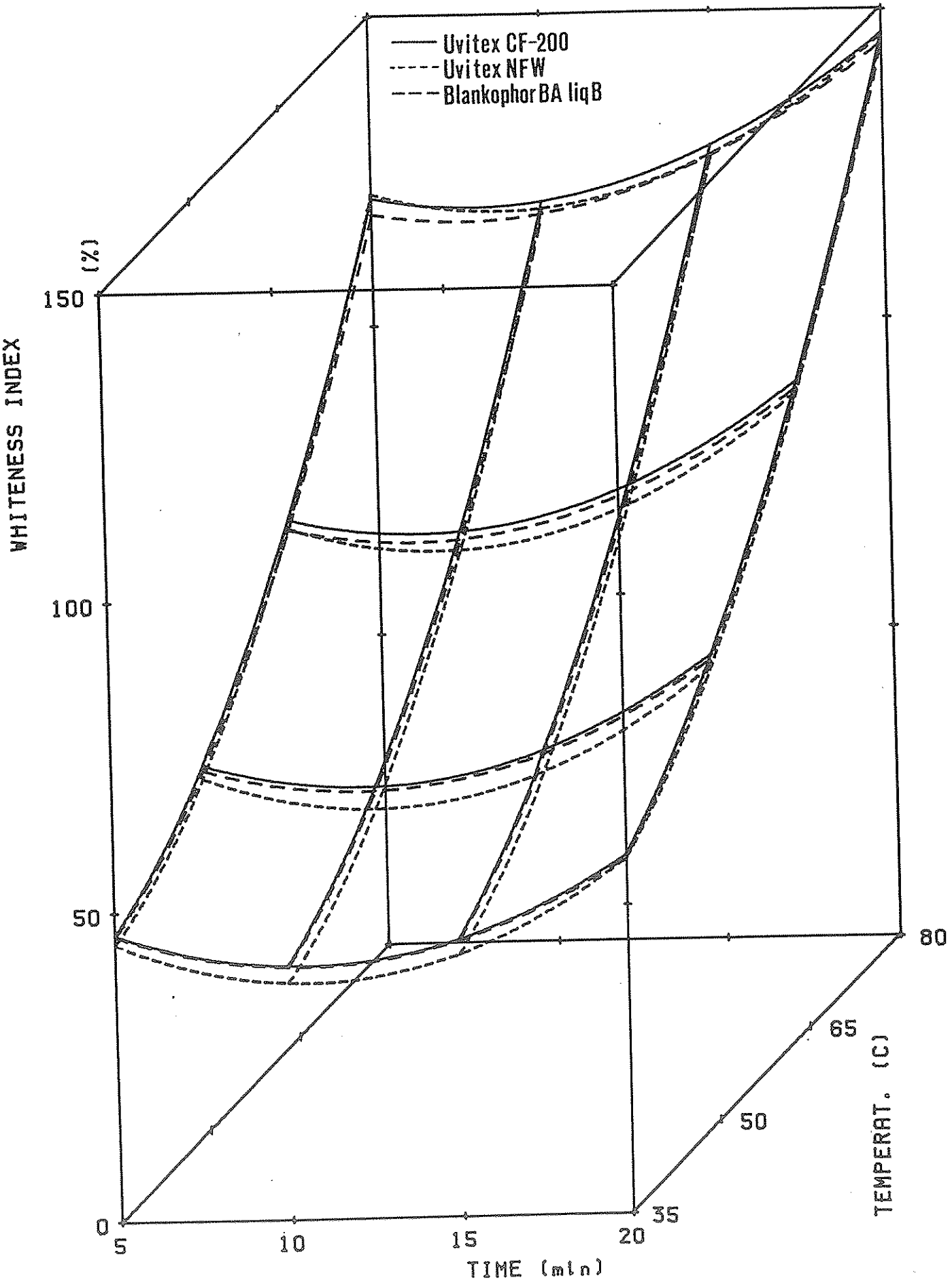
Uvitex CF-200
Uvitex NFW
Blankophor BA liqB
WHITENESS INDEX (%)
150
100
50
0
5
10
15
20
TIME (min)
35
50
65
80
TEMPERAT. (C)

FIGURE 5

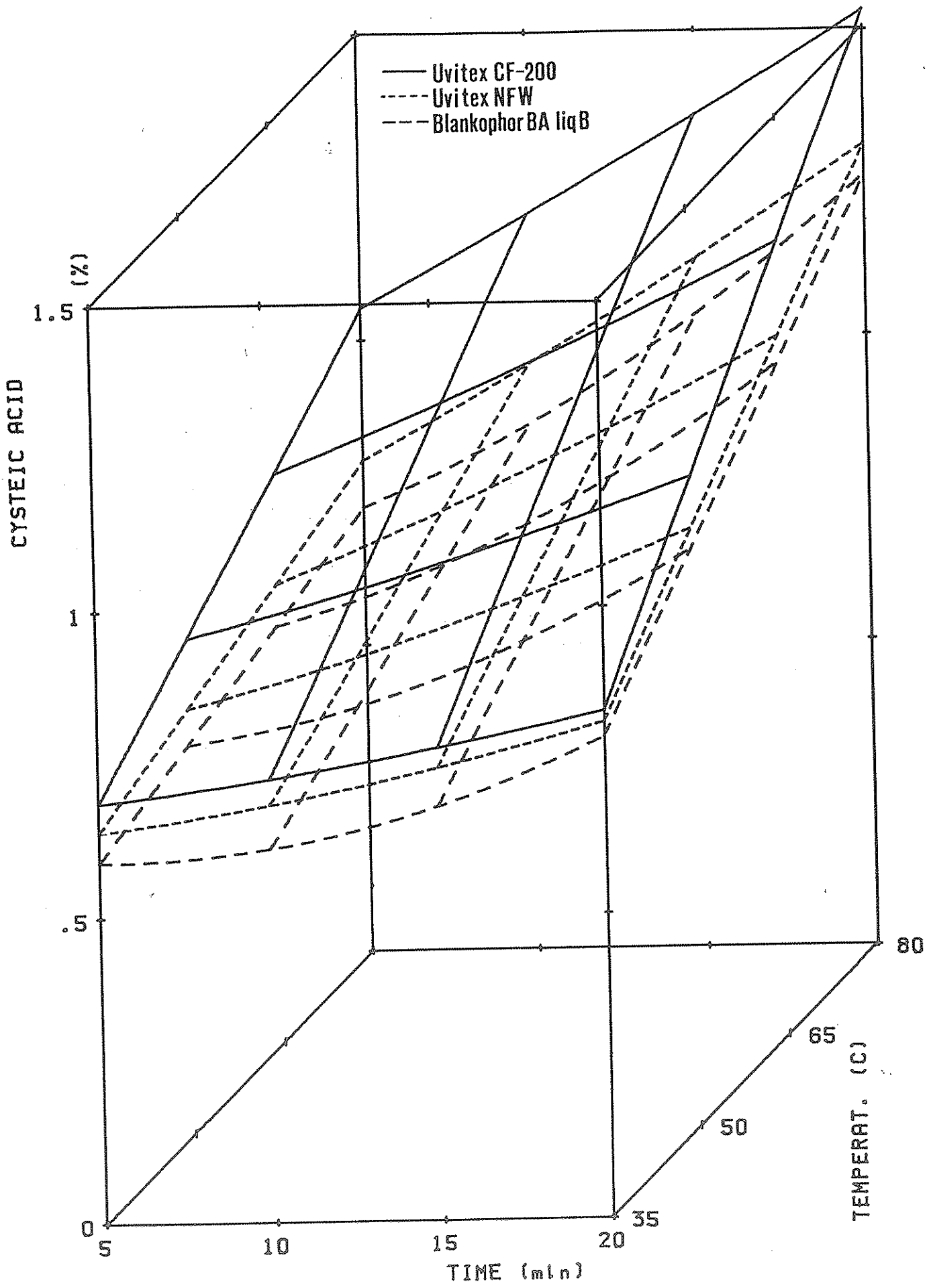
Uvitex CF-200
Uvitex NFW
Blankophor BA liq B
CYSTEIC ACID (%)
1.5
1
.5
0
5
10
15
20
TIME (min)
35
50
65
80
TEMPERAT. (C)

FIGURE 6

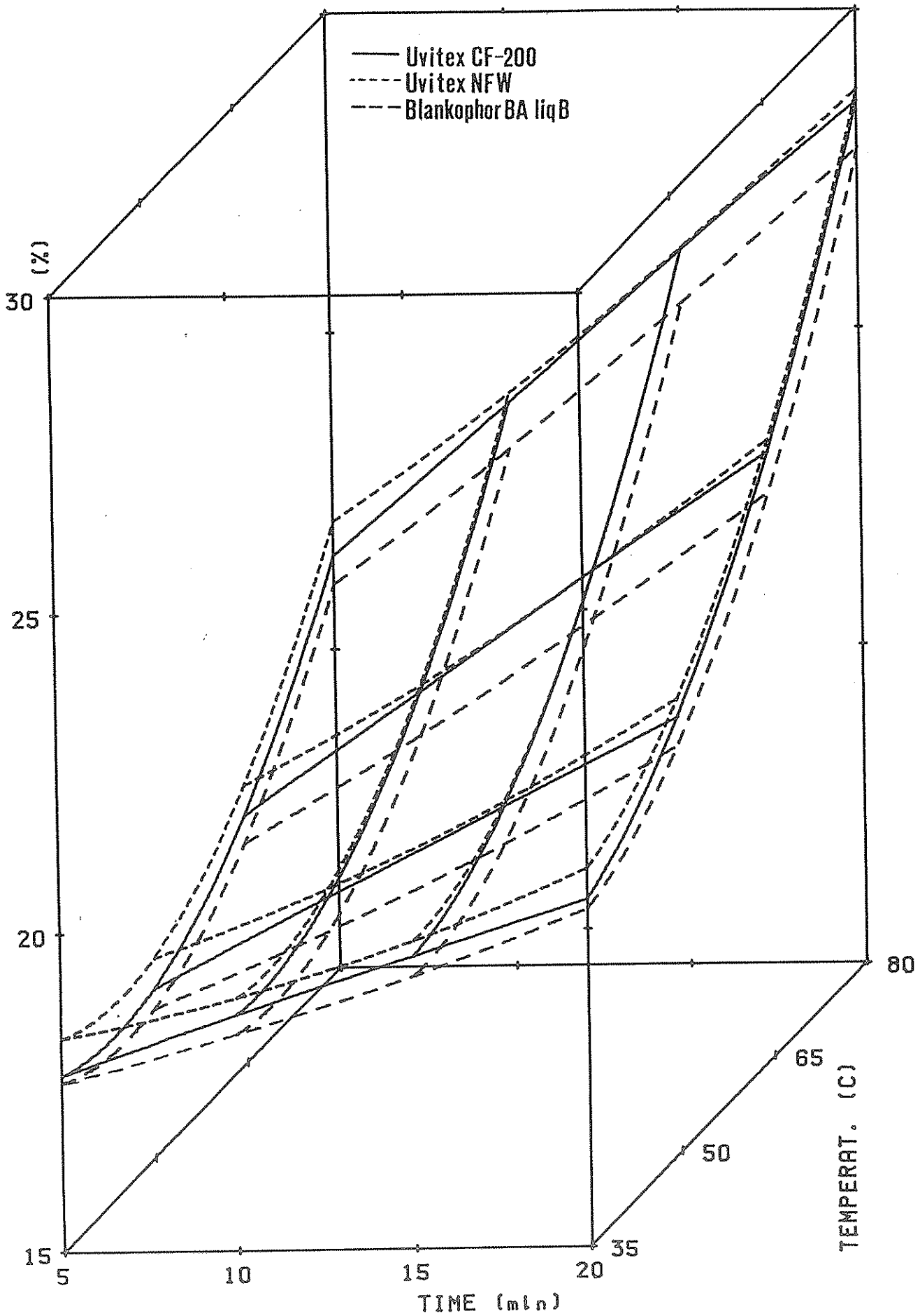
Uvitex CF-200
Uvitex NFW
Blankophor BA liq B
(%)
30
25
20
15
5
10
15
20
TIME (min)
35
50
65
80
TEMPERAT. (C)

FIGURE 7