

A correlation of the thickness of the nitrocellulose films prepared using a spinning disk technique with the observable properties of nitrocellulose solution

Vishnu S. Nadkarni[§], Sunil S. Bhagwat[¶] & Shriniwas D. Samant^{**}

[¶]Organic Chemistry Research Laboratory, [¶]Chemical Engineering Division

University Department of Chemical Technology
University of Mumbai,

Matunga, Mumbai 400 019, India

Received 10 June 1998; revised 15 September 1998

Spin coating method has been successfully used to cast CN-polyester composite films. Data have been presented after a systematic study of the nitrocellulose film formation by this technique by using various types of solvents. The method can be extended to prepare CN-polyester composite solid state nuclear track detector films useful for environmental dosimetry.

The use of nitrocellulose (CN) films as solid state nuclear track detectors (SSNTDs) for revealing the damage trails of ionizing radiations is well known¹. Some simple, but crude techniques have been used to prepare these films on a laboratory scale²⁻⁵. Thin CN films of 6-12 μm thickness are commercially available as SSNTDs. In the preparation of such films, besides arriving at the appropriate composition of the ingredients, it is very much essential to control the film thickness. The spinning disc technique, which was initially used in the semiconductor industry, was found to be a suitable technique for casting thin films of organic scintillating materials^{6,7} and different photoresists⁸. Theoretical studies on the flow patterns of viscous liquids on the spinning disc have been done and measurements of profile and film thickness have been reported^{9,10}.

Recently, we have reported the use of this technique for the preparation of thin CN films on a polyester substrate to form composite CN based SSNTD films^{11,12} and highlighted its importance in the nuclear track detection and the environmental dosimetry. This technique is also found to be highly useful for the batchwise preparation of thin films of plastic materials, and CN based SSNTD films, in particular, on a small scale. In the present paper we would like to discuss the effect of various process parameters on the thickness of the CN films.

Materials and Methods

Commercially, CN is available in different grades based on the viscosity. The viscosity roughly parallels the molecular weight of CN. Hence, these grades also reflect the degree of polymerization (DP) of CN. The viscosity governs the film forming property of the material, while the nitrogen content governs the sensitivity of the film to the radiations. It is well known that CN of high nitrogen content i.e., 12% or above gives better efficiency in the charge particle detection. Thus, out of the different grades of commercial CN tested with respect to viscosity and typical film forming properties for SSNTD applications, the following two grades were selected for further study.

- (i) *Sample A*: (ICI, India, henceforth CN-I), grade - HX 30-50; viscosity - $\frac{1}{2}$ s and nitrogen content - 11.9 % and
- (ii) *Sample B*: (Asha nitrocellulose, India, henceforth CN-A), grade - ES 30/40 s; viscosity - 30-40 s; nitrogen content - 12.2 %.

The spin coating was done using a Flood Spinner (Indo-German Engineers, Bombay) prepared as per our specifications and had a fixed rpm of 3000 at 230 V, 50 Hz AC power. It had a provision for holding a tinned mild steel plate of size 15 cm x 15 cm by means of a magnetized chuck and a presettable timer to control the time of the rotation of the disc. Figure 1 shows the

[§]Present address: Department of Chemistry, Goa University, Goa 403 207, India

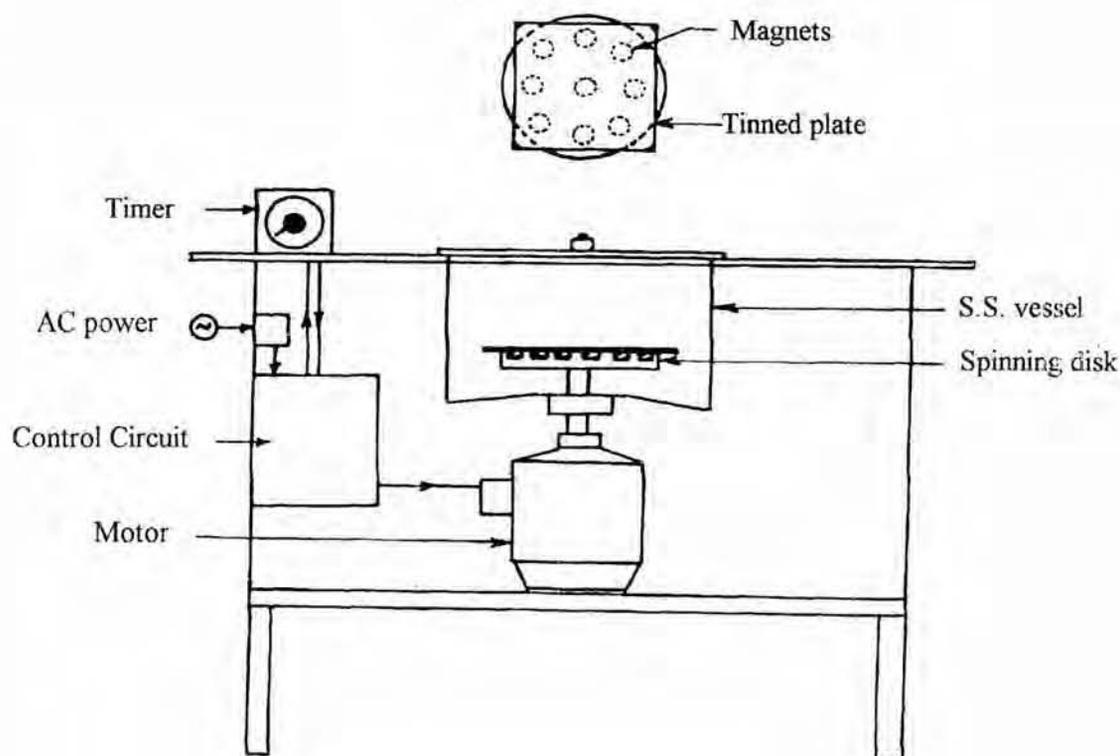


Fig. 1 - The flood spinner

schematic diagram of the flood spinner. The film thickness measurements were carried out using a digital thickness gauge, Deltameter DR (Electronic Enterprises, Bombay) with an accuracy of $\pm 5\%$.

Determination of the viscosity

Commercially available CN was carefully dried in air for four days. 40 ml solutions of concentrations 10, 15, 20, 25 and 30% (w/v) were prepared using air dried CN in the given solvent. The solutions were allowed to stand for 30 min in stoppered test-tubes so as to minimize the air bubbles present. In the case of a binary solvent, a 1/1 (v/v) mixture of the two solvents was used to prepare the CN solutions as above. The viscosities of the solutions were determined using a Ostwalds U tube viscometer, calibrated with 98% glycerol of known viscosity. The densities of the CN solutions were determined by using a specific gravity bottle.

Preparation of the CN films by spin coating

A commercially available 100 μm thick polyester film of size 15 cm x 15 cm was fixed to the tinned plate by means of cellotape at the corners. 10 ml CN solution of a given concentration was prepared in a solvent under study as mentioned above and left undisturbed for 30 min in a stoppered test tube. The solution (3 ml) was then poured at the centre of the disc over the polyester

base film attached to the tinned plate and the disc was spun for 1.5 min, when a flat, thin, uniform CN film was obtained over the polyester base. The method can be extended to prepare SSNTD CN films of required thickness (generally 10-12 μm) by selecting suitable solvent and additives like plasticizers and dyes. The reproducibility of this method was tested by many batches of the detector films.

Determination of the thickness of the CN film

The thickness of the polyester base (size 15 cm x 15 cm) was noted over randomly selected 30 points, followed by a similar measurement of thickness after coating the polyester base with CN solution. The difference in the two average thicknesses gave the thickness of the CN film. The average film thickness values have been rounded off to the nearest integer.

Results and Discussion

Viscosity of CN-I solutions in pure solvents

As CN is highly soluble in lower esters, ketones and cellosolves, a few solvents from each category were selected for the viscosity study; the selection was also based on the easy availability on large scale. Following

Table 1- Viscosities of the CN-I solutions in various esters, ketones and cellosolves

Conc. of the solution (w/v)	Viscosity (cP, 30°C)						
	EA	BA	AA	MEK	MIBK	MC	EC
10	53	85	95	25	61	124	148
15	141	354	359	99	258	422	537
20	169	1084	955	394	848	1266	1441
25	1337	2255	2475	704	1926	2310	3063
30	3517	4010	5339	1563	4750	5275	5434

Table 2- Viscosities of CN-I solutions in (methyl/ethyl cellosolve + ethyl acetate), (methyl/ethyl cellosolve + methyl ethyl ketone) and (n-butyl/isoamyl acetate + ethyl acetate) mixtures.

Conc. of the solution (w/v)	Viscosity (cP), 30°C					
	EA + MC	EA + EC	MEK + MC	MEK + EC	EA + BA	EA + AA
10	72	89	57	67	65	64
15	251	269	213	260	249	233
20	828	873	586	727	849	620
25	1212	1437	1120	1470	1893	1338
30	2692	3120	2380	3468	2876	2316

solvents were used for the present study: Esters : ethyl acetate (EA), *n*-butyl acetate (BA), isoamyl acetate (AA); ketones : methyl ethyl ketone (MEK), methyl isobutyl ketone (MIBK); and cellosolves: 2-methoxyethanol (methyl cellosolve)(MC), 2-ethoxyethanol (ethyl cellosolve) (EC)

Out of these BA, AA, MIBK, MC, EC are high boiling solvents, while the remaining are low boiling in nature. The viscosity of CN-A solutions in various solvents are given in Table 1. It was observed that around a viscosity of 1500 cP, corresponding to 15-20 % concentration, the solution became very viscous and was difficult to pour in the medium bore U tube viscometer (No. BS 4,165). Hence, for higher concentrations, a wide bore U tube viscometer (SCAM No.7) was used. It worked satisfactorily up to about 30 % concentration of CN-I, corresponding to viscosity of about 4000 cP. Due to this limitation, the present study was done only up to 30 % (w/v) concentration.

It was also observed that, in each category of the solvents, the viscosity increased with the increase in the molecular weight of the solvent. Thus, amongst the solvents selected, at any concentration, the viscosity of the solution increased in the order - ketone < ester < cellosolve.

Viscosity of CN-I solution in mixed solvents

As the viscosity is one of the main factors governing the thickness of the spin casted film, it may become imperative to build up the viscosity to the desired level at the given concentration of CN-I. Hence, we decided to study the viscosity of the CN-I solutions in binary mixtures of the above solvents. Mixtures of high boiling and low boiling solvent were selected as follows: cellosolve + ethyl acetate; cellosolve + methyl ethyl ketone; and high boiling ester + ethyl acetate. Table 2 records the viscosities of the CN-I solutions in these mixtures.

Table 3- Characteristics of some solvents, and viscosity of 30% CN-I solution in the respective solvents at 30°C

Solvent	Characteristics of solvent			Viscosity of 30% CN-I solution(cP)
	Boiling point (°C)	Molecular weight	Solubility parameter (cal/cm ³) ^{1/2}	
EA	78	88	9.1	3517
MEK	80	72	9.3	1503
MIBK	110	100	8.6	4750
BA	125	116	8.5	4010
AA	135	140	7.8	5339
MC	165	76	11.4	5275
EC	140	90	10.5	5434
cellulose(di)nitrate	-	-	10.55	-

Table 4- The average CN-I film thickness in the case of CN-I solutions in some esters, ketones, cellosolves and mixtures of solvents at different concentrations

Conc. of the soln. (w/v)	Average film thickness (µm)										
	EA	BA	AA	MEK	MIBK	MC	EC	EA + MC	EA+ EC	MEK + MC	MEK+ EC
10	1	1	2	3	1	1	1	3	2	1	1
15	4	2	3	3	3	1	1	4	4	2	2
20	7	4	5	5	5	3	2	6	6	4	6
25	12	7	8	7	8	6	4	9	11	7	8
30	13	9	12	18	14	7	6	13	14	12	15

When the mixtures of EA and cellosolve were used it was found that at any concentration the viscosity was lower than the viscosity in the cellosolve, but in the lower concentration region (< 20%) it was higher than the viscosity in EA at equal concentrations. A similar trend was also observed in the case of the mixtures of MEK and cellosolves and the mixtures of high boiling esters and EA.

Solubility parameter study

To explain the dependence of viscosity on the nature of the solvent, an attempt was made to relate the viscosity with the solubility parameter of the solvents. Table 3 lists the solubility parameters of different solvents and the viscosities of 30 % CN-I solutions in the respective

solvents, along with the boiling points and the molecular weights of the solvents.

A liquid acts as a good solvent for a solute, when the two are compatible and can coexist in molecular state with a minimum tendency of separation. The compatibility may be expressed in terms of the solubility parameter. The two substances are said to be compatible, if their solubility parameters are close to each other. Thus, a polymer will dissolve in a solvent having solubility parameter closer to that of the solvent. The solubility parameter of CN is 10.55 which is closer to that of cellosolves which lie in the range 10.5 to 11.4. Thus, cellosolve is a 'true solvent' for CN. Such a solvent overcomes the cohesive forces in the polymer chains due to the strong attractive forces between the solvent and

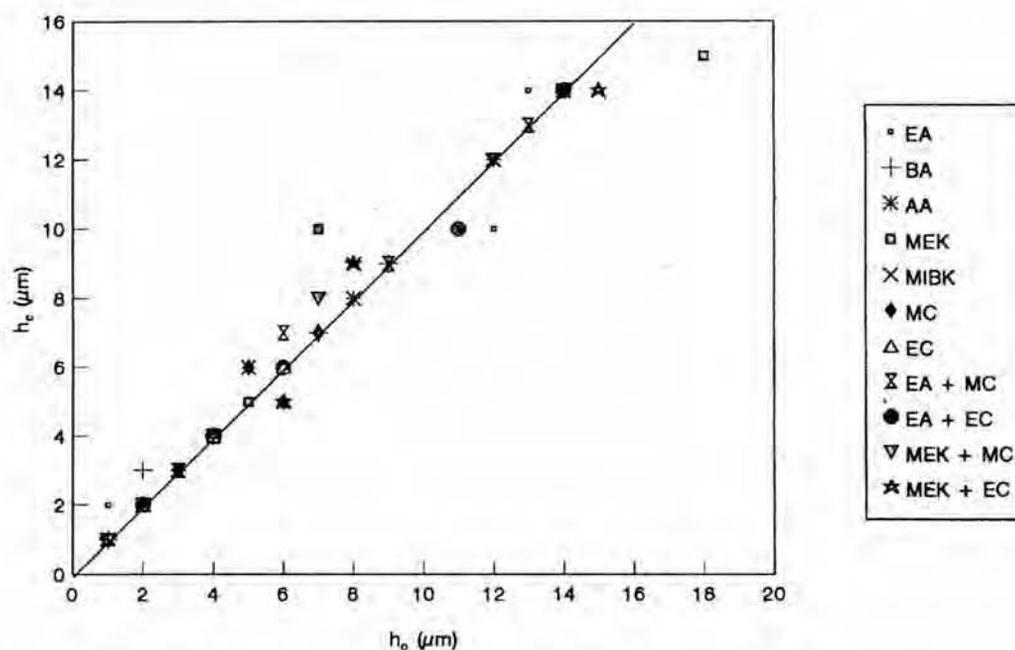


Fig. 2- Parity plot showing the observed film thickness against the calculated film thickness for the modified Damon equation of the type $y = mx + c$

Table 5- CN-I film thickness at constant viscosity or at constant concentration in different solvents

Sr. No.	Solvent	Concentration of solution (w/v)	Viscosity at 30°C (cP)	Film thickness (μm)
(a) At constant concentration				
1	Methyl cellosolve	25	2310	6
2	Ethyl acetate	25	1337	12
3	MIBK	25	1926	8
4	MEK + EA in 1:1	25	1212	9
(b) At constant viscosity (interpolated values)				
1	Methyl cellosolve	25	2500	5
2	Ethyl acetate	28	2500	13
3	MIBK	26	2500	9
4	MEK + EA in 1:1	30	2500	12

the polymer chains. The polymer chains then are uncoiled and are more and more surrounded by the solvent molecules giving a more viscous solution.

Study of the effects of viscosity and concentration of the CN-I solutions on the film thickness

CN-I solutions were prepared in different solvents at different concentrations and the corresponding CN-polyester composite films were prepared. Table 4 shows the

average film thickness of the films prepared using different solvents at different concentrations.

As has been mentioned earlier, the concentration-viscosity balance can be manipulated as desired by taking suitable solvent mixtures. Hence, we felt it worthwhile to prepare the films using the above mentioned solvent mixtures. Table 4 shows the average film thickness of the films prepared using different solvent mixtures.

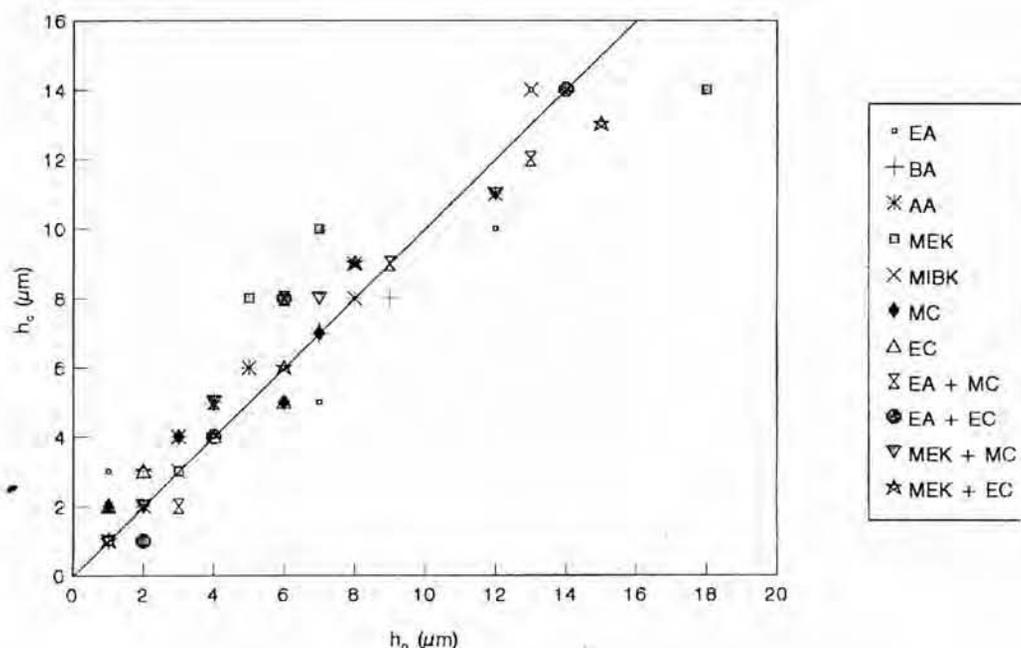


Fig. 3 - Parity plot showing the observed film thickness against the calculated film thickness for the modified Meyerhofer equation of the type $y = mx + c$

Correlation of the film thickness with some observable properties of the solvents

A theory has been developed to correlate the film thickness with the viscosity of photoresist solutions for the spin coating technique^{6,9,10}. Damon proposed Eq.(1) which relates the film thickness (h) with the concentration (c) of the solution and the spinning speed (f).

$$h = K_1 \times c^2 / f^{0.2} \quad \dots(1)$$

where K_1 is a constant characteristic of the solvent-polymer system used⁷.

Alternatively, Meyerhofer proposed Eq. (2) which relates the film thickness (h) to the viscosity (V) of the solution and the spinning speed (f).

$$h = K_2 \times V^{0.1} / f^{0.2} \quad \dots(2)$$

where K_2 is a constant characteristic of solvent-polymer system used⁸.

The above two equations have a major limitation that they are applicable only for a single solvent-polymer system, and cannot be generalized for a variety of solvents. For example, one would not get the same thickness - (a) by keeping the concentration of the solution fixed, but by changing the solvent, as per Eq. (1) or (b) by keeping the viscosity of the solution fixed, and changing the solvent, as per Eq. (2). This can be highlighted from the data we have gathered in Table 5.

It is clear that, in spite of the constant concentration or viscosity of the solution, there are large variations in the film thickness, depending upon the nature of the solvent used.

In a model experiment, 30 % CN-I solution was prepared in EA (b.p. 78°C) and EC (b.p. 140°C). From each solution, films were prepared by spinning the disc for 0.5, 1 and 1.5 s. For each setting, there was a delay time of 7 s and a deceleration time of about 30 s, over which the speed decreased to zero.

After 0.5 s, it was observed that both the solutions spread uniformly over the disc and the radius of the circular film in both the cases was about 9 cm. EC gave a thicker film of about 41 μm , while EA gave a thinner film of 31 μm thickness. When the disc was spun for 1 s, the solution spread further and covered the entire disc area (15 cm x 15 cm) and the film thickness was reduced; EC gave a film of 6 μm thickness and EA gave a film of 12 μm thickness. Though in normal practice we spin the disc for 90 s, the actual film formation takes place within the first second itself. After spinning the disc for 0.5 s, it was found that the EC film was wet, while the EA film was almost dry. The same observations were noted after 1 s. After spinning for 90 s, the EC film dries completely.

At this point, considering the above observations, we visualized the following model for the formation of a thin CN-I film over the spinning disc. When the disc is

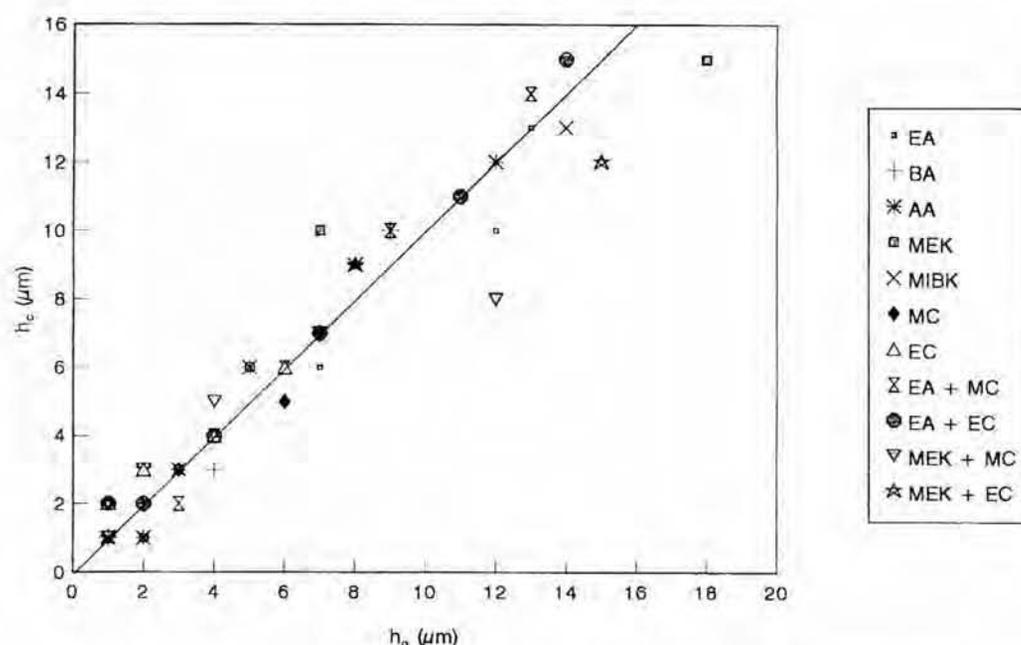


Fig. 4 - Parity plot showing the observed film thickness against the calculated film thickness using Damon equation in the form $y = mx$

spun at a constant speed, the solution on the disc is subjected to two processes -

- (1) centrifugal force acting on the solution, pushing it radially out and spreading it uniformly on the disc, and
- (2) evaporation of the solvent leading to the deposition of the solid mass on the disc.

In the above experiment, the two solvents namely EA and EC have almost the same density (0.902 and 0.930 g/cm³ respectively). Obviously, both the CN-I solutions have the same density. Thus, on the spinning disc the solution would experience the same centrifugal force. This is supported from the fact that both the solutions were spread on the spinning disc to the same extent when spun for a fixed time (0.5 s). The other process, i.e., the evaporation of the solvent is assisted by the spinning of the disc. A low boiling solvent is expected to evaporate faster than a high boiling solvent and hence, the CN solution in a low boiling solvent would deposit more solid per unit area than the solution in a high boiling solvent during the radial movement of the solution on the disc. This explains the formation of a thicker film in the case of a low boiling solvent than the film formed using a high boiling solvent. This also explains the importance of the solvent in this technique.

Correlation analysis

We have previously seen that Eqs (1 and 2) have been proposed to correlate the film thickness in the case

of solutions of photoresists. An attempt was made to apply these equations to the CN-solvent systems for the first time.

It was essential to find out whether the relationship between h and c^2 or $V^{1/3}$ is of the type ($y = mx$) or ($y = mx + c$). A correlation analysis was performed to test the applicability of these equations to various nitrocellulose solvent system.

The data obtained were evaluated in light of these two equations in order to see whether the data followed a linear behavior and whether the line passed through the origin. The observed film thickness (h_o) was plotted against the variable terms in the two equations, i.e., $c^2/f^{1/2}$ and $V^{1/3}/f^{1/2}$, respectively, and the corresponding slopes (i.e., K_1 and K_2) were calculated as $(\sum y/\sum x)$. Using these values of K_1 and K_2 , film thicknesses were recalculated at each concentration. Graphs were plotted showing the variation of these calculated film thickness (h_c) and the observed film thickness (h_o) against the terms c^2 and $V^{1/3}$ for the two equations. From the two graphs, the goodness of fit was evaluated as $\sum (h_o - h_c)^2$. It can be seen that, the Damon equation correlates the film thickness values better as compared to the Meyerhofer equation.

Next, the data were correlated with the equation $y = mx + c$. Hence, Eqs (1 and 2) may be modified as follows. For Damon equation,

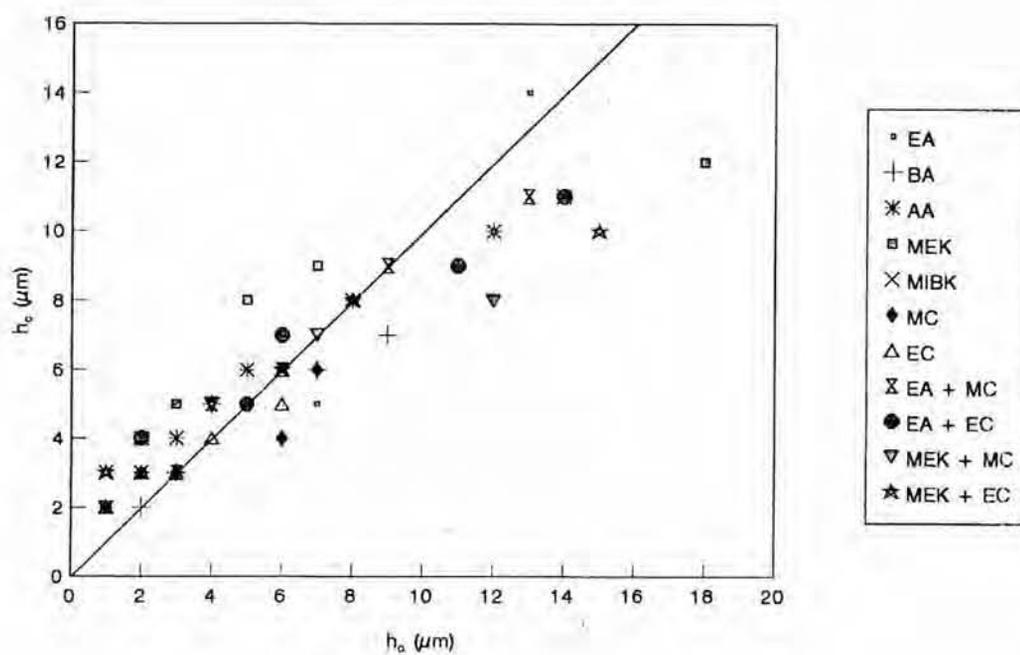


Fig. 5 - Parity plot showing the observed film thickness against the calculated film thickness using Meyerhofer equation in the form $y = mx$

Table 6- Results of correlation analysis

Solvent	Modified Damon equation $y = mx + c$				Modified Meyerhofer equation $y = mx + c$			
	K_1	c_1	$\sum(h_a - h_c)^2$	$\text{corr}(x,y)$	K_2	c_2	$\sum(h_a - h_c)^2$	$\text{corr}(x,y)$
EA	8610	0.328	7.62	0.964	54.4	-0.691	16.9	0.919
BA	5410	0.344	0.87	0.989	35.9	-1.937	3.03	0.962
AA	6680	+0.701	1.31	0.989	41.3	-1.713	2.35	0.981
MEK	9670	-0.674	18.4	0.935	87.7	-3.998	28.6	0.896
MIBK	8780	-0.801	2.76	0.987	55.5	-3.518	2.87	0.987
MC	4620	-0.190	1.04	0.983	30.4	-2.380	1.24	0.979
EC	3510	+0.169	0.35	0.990	22.4	-1.589	1.11	0.967
EA + MC	8310	+0.430	1.57	0.992	65.2	-3.782	5.49	0.971
EA + EC	6780	+1.576	1.97	0.984	54.5	-1.724	6.93	0.944
MEK + MC	7960	-1.325	1.27	0.993	67.5	-5.121	3.92	0.977
MEK + EC	9240	-1.371	2.70	0.989	68.4	-5.254	3.20	0.986

Table 7- The viscosity of CN-A solutions different and the CN-A film thickness in various solvents

Concentration (%)	Viscosity (cP), 30°C			CN-A film thickness (µm)	
	EA	EC	EA + EC	EA	EA + EC
2	15	24	30	1	1
4	121	189	175	1	1
6	577	729	850	2	1
8	1574	2097	2535	4	2

Table 8- Results of correlation analysis for CN-A solutions

Solvent	Modified Damon equation				Modified Meyerhofer equation			
	y = mx + c				y = mx + c			
	K_1	c_1	$\sum(h_o - h_c)^2$	corr(x,y)	K_2	c_2	$\sum(h_o - h_c)^2$	corr(x,y)
EA	28600	0.124	0.20	0.983	18.3	-0.602	0.45	0.961
EA + EC	15700	-0.142	0.10	0.971	9.00	-0.589	0.08	0.978

$$h_o = K_1 \times c^2 / \Gamma^{1/2} + c_1 \quad \dots(3)$$

where, c_1 is the intercept of the line. Similarly, Meyerhofer equation becomes -

$$h_o = K_2 \times V^{1/3} / \Gamma^{1/2} + c_2 \quad \dots(4)$$

In this case, the constants K_1 and K_2 , i.e., slopes of the plots of the observed film thickness (h_o) against the terms $c^2/\Gamma^{1/2}$ and $V^{1/3}/\Gamma^{1/2}$ respectively, and the intercepts were determined with the help of a computer program. The predicted film thickness values (h_c) and the observed values (h_o) were plotted against the terms c^2 and $V^{1/3}$ for the two equations, respectively. From the two graphs the goodness of fit was evaluated as $\sum (h_o - h_c)^2$. The correlation coefficient $corr(x,y)$ was also determined using the computer program. Considering the goodness of fit values and correlation coefficients for Eqs (1 and 2) with and without intercept for various solvents, it was found that the modified Damon equation, $h_o = K_1 \times c^2 / \Gamma^{1/2} + c_1$ was more suited to correlate the data (Table 6).

The $c/V - h$ relationships were studied using nitrocellulose solutions in various solvents like esters, ketones, cellosolves and mixed solvents. The goodness of fit val-

ues for $y = mx + c$ type relation derived for the Damon equation were found to be the smallest for all the above systems and the correlation coefficients closest to unity, indicating a better fit between the calculated and the observed values within experimental error. The parity plots (Figs 2 - 5) also indicated that the goodness of fit was better with the Damon equation as compared to the Meyerhofer equation.

It appeared from the data that, both these equations showed larger deviations when applied to low boiling solvents, i.e., ethyl methyl ketone or ethyl acetate, compared to the high boiling solvents. The deviations observed in the case of Meyerhofer's equation may be attributed to-

- (1) Change in the rate of evaporation of the solvent, as concentrated CN-I solutions were used. It is known that, a CN solution on standing form a dry CN film over the surface which prevents further evaporation of the solvent. In the case of spin coating also, the drying would take place from the top to the bottom. Thus, the topmost CN layer which is dry, would prevent the rapid evaporation of the solvent and the thickness of wet CN mass is reduced considerably due to the action of the centrifugal force.
- (2) Ostwald viscometer method, though is suitable for

the use in the laboratory due to its simplicity, cannot be regarded as an accurate method in the case of concentrated CN solutions.

- (3) Nitrocellulose used may not have a uniform molecular weight distribution. The accurate weighing of CN sample is also not possible due to the presence of a damping medium like butanol.

We also tried to correlate the CN film thickness for the other grade of nitrocellulose i.e., CN-A. As the behaviour of the CN solution over the flood spinner was known previously, the variation of viscosity was studied in three different solvents only, viz., ethyl acetate, ethyl cellosolve and a mixture of ethyl acetate + ethyl cellosolve (1/1, v/v). It was also observed that the solubility of CN-A in these solvents was less as compared to CN-I. Hence, a longer time was required to dissolve CN-A in these solvents and make homogeneous solutions. In this case, it was found that, in any of the above mentioned solvents the viscosity could be measured only upto a concentration of 8%. At 10% or higher concentrations, filling the viscometer with the CN-A solution was found to be very difficult. The viscosities of CN-A solutions in different solvents at different concentration in the range 2-8% are shown in Table 7.

The films were cast using a flood spinner in the above mentioned solvents. Film thicknesses of CN-A in different solvents at different concentrations are given in Table 7. From Table 7, it can be seen that though the viscosity of the CN solution at 8% concentration was very high, it gave a film of only 2-4 μm thickness. This observation was strikingly different as compared to the observations in the case of CN-I. The same problem was noted especially in the case of ethyl cellosolve, even when 8% solution of CN-A was spun over the flood spinner. It was found to be very difficult to note the thickness of CN film formed over the polyester base.

The correlated values of the slope K , i.e., constant in the Damon equation ($h = K \times c^2 / f^{1/2}$), was found out (Tables 8) and hence, the concentration of CN-A solution required for preparing a film of thickness 10 μm was calculated. It was found to be 18%. Therefore, a 18% solution of CN-A was prepared in ethyl acetate + ethyl cellosolve (1/1, v/v) and was allowed to stand for about 2 h in a stoppered test tube and spun over the spin-

ner to get a composite film. The thickness of the film was found to be between 10-11 μm . It was observed that, in this film (15 cm x 15 cm), the corners had strong contours, due to the very high viscosity of the solution.

When K_1 in the case of the Damon equation for CN-A was compared with that for CN-I, it was seen that its value was almost double, i.e., $K_{\text{CN-A}} / K_{\text{CN-I}} = 2$. This seemed to be due to the increase in the molecular weight of CN-A. Thus, it appeared that the constant K is a function of the molecular weight of the polymer (CN) as well as that of the nature of the solvent.

The method was extended to cast nitrocellulose SSNTD films (stripping and non-stripping type) by using solutions of CN in suitable solvents like EA or (EA+MC), containing suitable dyes and plasticizers.

Acknowledgement

The authors are grateful to the Atomic Energy Regulatory Board, Government of India, for a research grant and a fellowship to VSN.

References

- Collver M M, Benton E V & Henke R P, AEC accession No. 4251 (1965) USNRDL-TR-917, (C.A. 65: 11719 f)
- Benton E V (1968) USNRDL-TR-68-14
- Lupica S B, *Radiochem Radioanal Lett.*, 21(1976) 31-38
- Bhagwat A M, Deo V R & Soman S D, Report-Bhabha Atomic Research Centre Bombay, BARC/ I-413 (1976)
- Bhagwat A M & Soman S D *Proc Int Congr Int radiat Prot Assoc*, 3rd, (Sept 1973) 2(CONF-730907-P2) (Pub 1974) pp 852-856
- Norbeck E, Dubbs T P & Sobotka L G *Nucl Instr Methods Phys Res, Sect A*, A 262(1987) 546-547
- Damon G F, *Proc Kodak Seminar on Microemulsion*, 2nd, Rochester, N York, (1967) 36
- Meyerhofer D, *J appl Phys*, 49 (1978) 3393
- Emslie A G, Bonner F T & Peck L G, *J appl Phys*, 29(1958) 858
- Acrivios A, Shah M J & Petersen E E, *J appl Phys*, 39(1960) 963
- Raghunath B, Samant S D & Iyer M R, *Radiat Meas*, 24(1995) 95
- Nadkarni V S & Samant S D, *Radiat Meas*, 27 (1997) 505
- Brydson J A, *Plastic materials* (Butterworths), 4th ed. Chapter 5, (1976)