Effect of Process Parameters on Tenacity of Acrylic Fiber

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Abstract- Acrylic forms an important fibre in the synthetic fibre sector. Its use in the winter wear sector is increasing & applications in other sectors are also being tried out. The need for improvement in fibre characteristics becomes important in this context. In the present study an attempt has been made to assess the effect of various additives, dope concentrations and spinning conditions on the properties of the acrylic stable fibres. It is found that the type of additives, dope concentration, bath temperature and stretch ratio play major role in deciding the tenacity in protofibre and final fibre. Bath composition. Jet stretch. Drying temperature propile do not play major role in deciding the tenacity. Stretching bath temperature has effect on tenacity but higher temperature causes filament breakages.

Keywords – Acrylic Fiber, Tenacity, Co-agulation, Spinning, and Proto Fiber

I. INTRODUCTION

In present study following parameters were selected to study overall effect on protofibre as well as on final fibre properties.

Parameters under study are charted underneath.

Additives in dope:

- Dope concentration
- Coagulation bath composition & temperature
- Jet stretch
- Stretching temperature & concentration
- Drying conditions therefore dryer temperature profile.
- Dyeing behavior

Normal wet-spun fibre composed of various additives in polymer. In this study dope additives co-monomer as well as dyeing behavior. By varying concentration of dope, its overall effect on homogeneity of fibre structure, voids number, spinnability and bulk density of the fibre were studied. Coagulation bath composition & coagulation temp. Were varied in present study to study the effect on fibre process ability, bulk density, voids porosity & tensile properties. Jet-stretch ratio was also studied with reference to the performance of spinning & to see the effect on orientation of fibres. Effect of stretching temperature & concentration was studied to see the processibility, tensile properties & fibre structure.

Acrylic fibre is very different from other synthetic fibre like nylon & polyester. So a study was also carried out to evaluate effect of drying condition, therefore dryer temp. & speed profile, residence time & tension, on their crystalline structure, collapsing, crimping & dyeing behavior.

A comprehensive study was carried out on dyeing properties of final fibre with reference to rate of dyeing, depth also.

II. EXPERIMENTAL

The objective of present study was to assess the effect of spinning conditions on fibre processing and fibre properties of solution spun acrylic stable fibres. For this purpose it was proposed to use a complete laboratory model spinning line. However due to non-availability of complete equipment the experiments based on stretching to dryer were done on existing commercial spinning line and experiments based on co-agulation bath were done on laboratory model.

Therefore the scope of changing the various parameters had to be restricted. The product at various stages was tested for relevant properties.

A. DETAILS OF EXPERIMENTATION

Materials

The composition of prepolymerised mixture is given below :

Folymix Composition (%)

ACN	MA	DMF	SAMPS	H20
34.05	3.38	60.05	0.260	2.26

Methods

The polymerization was carried out in an autoclave with following process parameters.

Polymerization Parameters

Reactor Parameters

- Residence time = 12 hrs
- Temp. (Degree C)

A	D	E	B	C
71.5	71.5	71.5	71.5	71.5

- Catalyst Concentration = 168.PPM.
- Dope Temp. = 65^{o}C
- Dope Pressure = 2.0 kg/cm^2
- RV = 1.778

The polymerised material was processed to through commercial line and also through laboratory model. The standard parameters are given below :

Spinning Parameters

- Dope concentration - 23%
- Coagulation Bath Temperature - 20^{o}C
- Coagulation Bath Concentration - 55:45
- Jet- Stretch - 1.05

Stretching Parameters

- Stretching Temperature - 97^{o}C
- Stretching ratio - 5.9

Washing Parameters

- Washing Temperature - 60^{o}C

Drying Parameters

Speed Profile

Drum	Speed (MPM)
Da	71.7
Db	71.0
Dc	65.0
Dd	59.4
D1	53.1
D2	45.6

D3	43.0
D4	42.6
D5	41.8
D6	43.7
D7	46.8
D8	50.0
D9	54.1
D10	53.4

Temperature Profile

Zone 1 - 128
Zone 2 - 135
Zone 3 - 137
Zone 4 - 164
Zone 5 - 164
Zone 6 - 158
Zone 7 - 157
Zone 8 - 125

The effect are evaluated by varying following parameters

Spinning Parameters

Dope Additives
MA - 8.5%, 7.5%
SAMPS - 0.5%, 0.8%

Dope Concentration
20%
23%
25%

Bath Composition
DMF : DMW
45 : 55
50 : 50
55 : 45
Jet Stretch
1.06
1.10

Stretching Parameters

Stretching Ratio
5.5
5.8
6.0
6.5
6.8

Stretching Temperature
90^{o}C
95^{o}C
100^{o}C

Drying Parameters

Profile ‘a’ :
120, 130, 130, 155, 155, 150, 150, 120
Profile ‘b’ :
130, 140, 140, 165, 165, 160, 160, 130
Profile ‘c’ :
140, 150, 150, 175, 175, 170, 170, 140

EXPERIMENTAL DESIGN

CHART I: EXPERIMENTAL DESIGN FOR LABORATORY MODEL

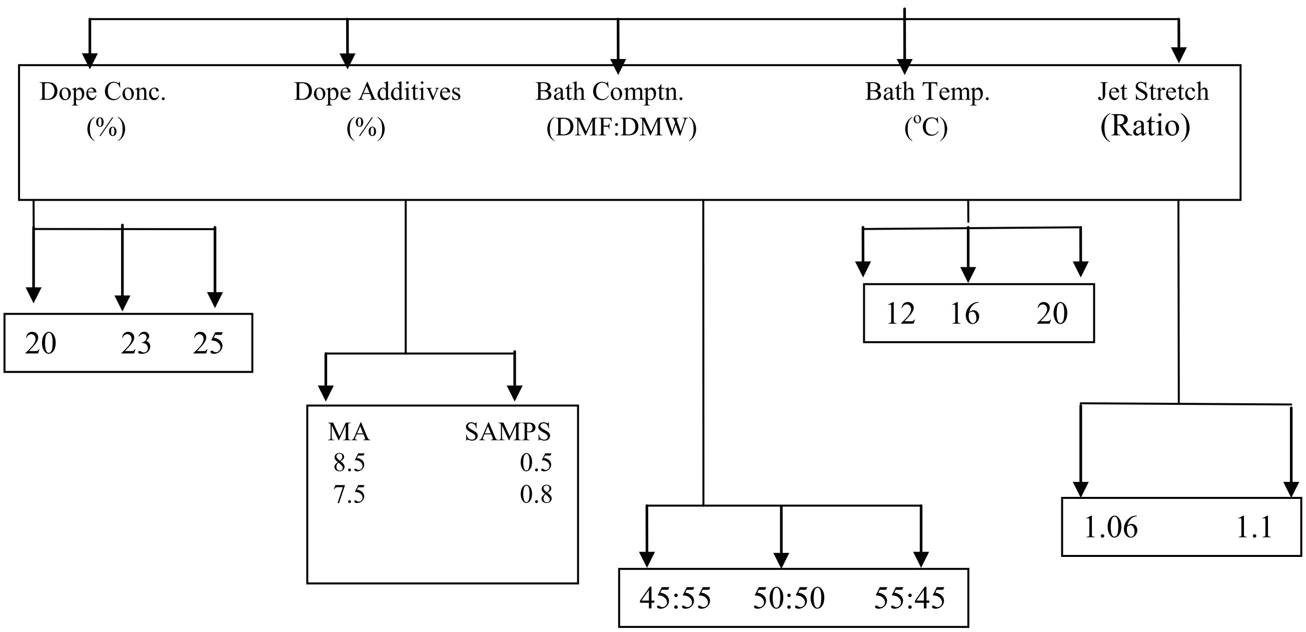

Chart-I gives the complete design of experiments done.

CHART II: EXPERIMENTAL DESIGN FOR COMMERCIAL LINE

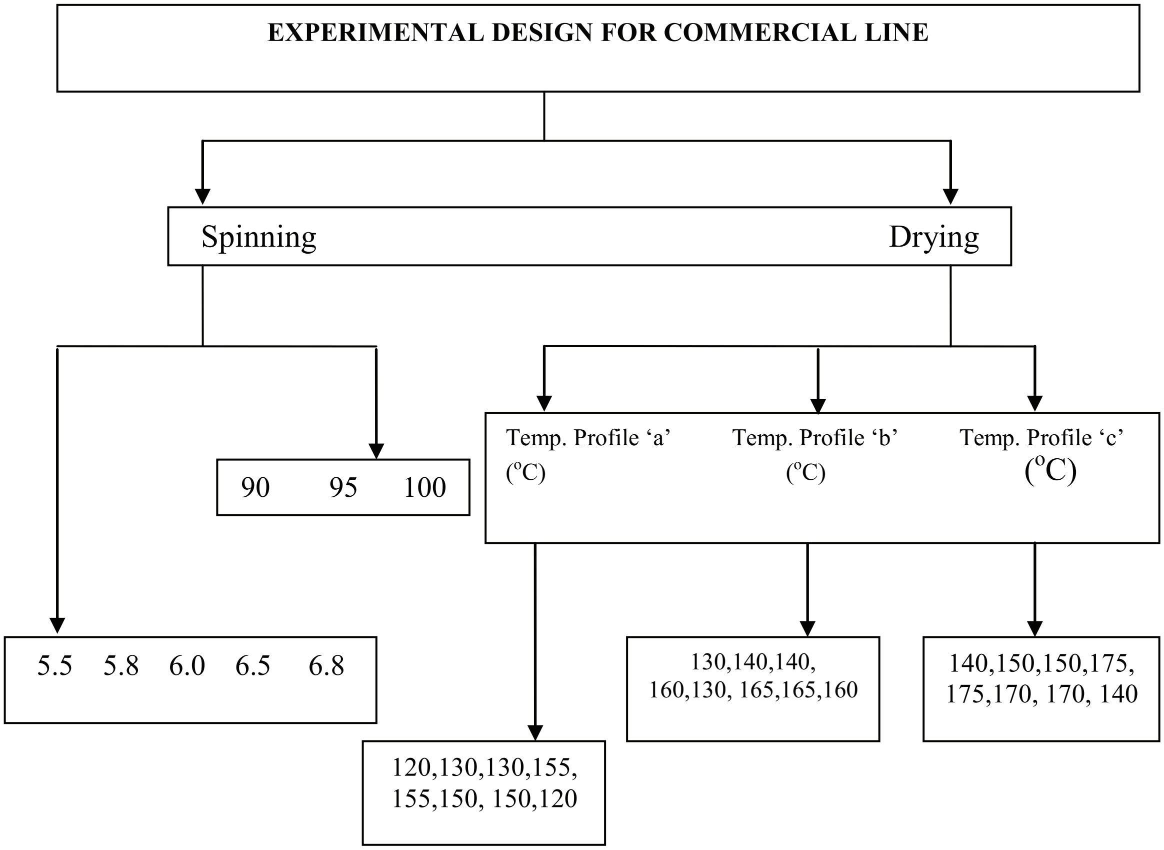

Chart- II gives the experimental design for commercial line.

EXPERIMENTS :

The samples at various stages were tested for the properties given below :

Co-agulation Stages

- Denier
- Density
- Voids/ C.S
- Void No
- Cross – Section

Stretching Stage

- Denier
- Tenacity
- Elongation
- Voids
- DMF %
- Crystallinity %

- Orientation Index
- Cross – Section

Drying Stage

- Denier
- Tenacity
- Elongation
- Dyeing Rate
- Dyeing Depth
- Crimps Stability
- Water Retention
- Crystalline %
- Orientation Index
- Cross – Section
- Whiteness Index

III. RESULTS AND DISCUSSION

The study relates to the assessment of

- Effect of Dope additives and concentration
- Effect of Co-agulation conditions
- Effect of stretching conditions
- Effect of drying conditions

The above conditions have varied and its effect on different properties is studied in detail.

EFFECT OF ADDITIVES IN DOPE AND DOPE CONCENTRATION

DOPE ADDITIVES

Dope additives like MA, SAMPS, TIO_2, and DMF affect the plasticity, dyeability, water retention, transparency, and solubility. Hence to study some of these behaviors, four types of polymer were prepared and spinning of above polymers were carried out under normal conditions. Fibres of these polymers were collected and evaluated for tenacity, Elongation, Dyeing properties and voids. The details of which are given in table-I.

TABLE-I DOPE ADDITIVES

Sl no.	Polymer composition(%)	Denier	Tenacity (gpd)	Elongn %	Voids (No./C.S)	C.S shape	Dyeing rate	Dyeing Depth
A	ACN - 90.5 MA – 8.5 SAMPS - 1	3.02	3.52	34.1	03	Kidney	Normal	Normal
B	ACN – 91.5 MA – 7.5 SAMPS – 1	3.04	3.65	28.3	08	No change	Slightly Reduced	Normal
C	ACN – 91.0 MA – 8.5 SAMPS– 0.5	3.05	3.53	32.3	03	No change	Very slow	Slightly Darker
D	ACN – 90.7 MA – 8.5 SAMPS – 0.8	3.04	3.57	33.9	04	No change	Slow	Darker

Reduction in percentage of co-monomer from 8.5 to 7.5 shows no change in denier and tenacity, slight reduction in elongation, and small increases in number of voids. It seems that by reducing the co-monomer, i.e. MA we are reducing the plasticity of fibre leading to decrease in elongation. There was no change in C.S. shape of fibre and remain kidney shape. As regard to change in dyeing co-monomer, i.e. SAMPS, we observed that increases or decrease in dyeing co-monomer has tremendous effect on rate of dyeing (graph for dyeing rate attached) as well as depth (dyed sample attached) of shade whereas no other physical parameters are affected.

DOPE CONCENTRATION

To a greater extent dope concentration influences the homogeneity of fibres. To study this effect with a normal polymer composition i.e 90.5% - ACN, 8.5% - MA, 1% - SAMPS were prepared with three different concentration of 20%, 23% and 25% and protofibre properties are assessed in terms of C.S, density and final fibre properties assisted in terms of tenacity, elongation, voids / C.S, density and Cross Section as shown in table

TABLE-II DOPE CONCENTRATION

		PROTOFIBRE			FINAL FIBRE				
Sr. no.	Polymer compo. & conc. (%)	C.S.	Voids / C.S.	Density	Tenacity	Elongn.	Voids/ C.S.	Density	C.S.
A	ACN – 90.5 MA – 8.5 SAMPS – 1 CONC. - 20	Normal	10	0.43	3.42	33.8	05	1.162	Normal
B	ACN – 90.5 MA – 8.5 SAMPS – 1 CONC. - 23	Normal	07	0.46	3.51	32.9	04	1.170	Normal
C	ACN – 90.5 MA- 8.5 SAMPS – 1 CONC. - 25	Normal	04	0.55	3.57	34.1	02	1.178	Normal

PROTOFIBRE

With all other factors held constant, increasing dope improves homogeneity which is evident from reduction of No. Of voids. No Simultaneous change in cross section of fibre was observed. However increase in density was observed due to reduced no. of voids with increase in solid concentration.

FINAL FIBRE

In terms of final fibre properties dope concentration shows an interesting trend in tenacity, no trend in elongation, reduction in voids and increase in density.

The increase in tenacity can be attributed to the reduction in no. Of voids resulting in improved homogeneity of structure and a better arrangement of molecules indicated by the increase in density was marked.

EFFECT OF COAGULATION CONDITIONS

The effect of coagulation conditions have been assessed in terms of bath compositions, temperature and jet stretch.

Bath Composition:

Spinning of a normal polymer was carried out with varying composition of DMF and water mixture i.e. 45 : 55, 50 : 50, 55 : 45, keeping bath temperature constant at a standard value of 16^{o}C. Protofibre and the final fibre were collected under these conditions and analysed for various parameters. The properties are given in table

CONCENTRATION

TABLE-III THE PROPERTIES BATH COMPOSITION

		PROTOFIBRE			FINAL FIBRE					
Sample	% DMF:WATER	Density G/cm^3	Voids (No.)	C.S.	Den.	Ten.	Elongn.	C.S.	Dyeing rate	Dyeing depth
A	45 : 55	0.43	09	Kidney shape	3.05	3.45	29.2	No change	No change	No change
B	50 : 50	0.45	06	Almost kidney shape	3.01	3.39	31.4	-	No change	No change
C	55 : 45	0.47	03	Almost kidney shape	3.08	3.48	30.7	-	No change	-

PROTO FIBRE

It is found that bath composition has a little effect on density of proto fibre as it is evident from the marginal change in density. This change is linked to the reduction of voids.

At the other hand the voids are reduced with increase of DMF concentration in bath. The photograph taken from surface scanning also shows continual reduction of voids with increase in bath concentration.

The cross section for different conditions remained the same.

FINAL FIBRE

In final fibre the denier, tenacity and elongation did not change markedly paving the way to conclude that the bath composition change does not influence above parameters practically. Cross – section, dyeing rate and dyeing depth also did not show any change. Even though smaller voids were removed by increasing the bath concentration the larger voids still remained in fibre structure which did not allow any remarkable change in further process.

Bath Temperature

Like bath composition bath temperature plays an important role in deciding the properties to be imparted in protofibre and subsequently in final fibre. So an experiment was done with a bath composition of 55 : 45, and temperature was varied as 12^{o}C, 16^{o}C and 20^{o}C. Protofibre and final fibre were analysed in terms of denier, cross section, tenacity, elongation and dyeing rate. It was studied for the same standard parameter, which were for bath concentration. Findings are summarized in table IV.

TABLE-IV FINDING OF BATH TEMPERATURE

		PROTO FIBRE			FINAL FIBRE						
Sample	Temp.	Denier	Voids	C.S	Denier	Temp.	Elongn.	C.S	Dyeing rate	Dyeing depth	Wrapping Tendency
A	12^{o}C	0.49	02	Kidney	3.06	3.62	28.2	Kidney	No chang	No chang	Normal

									e	e	
B	$16^{o}C$	0.47	04	kidney	3.08	3.45	31.1	kidney	-	-	Slightly Normal
C	$20^{o}C$	0.44	10	Slightly towards circular	3.03	3.39	32.3	Slightly towards circular	-	Very slightly better	Higher

PROTO FIBRE

From this study, we observed that with increase in bath temperature there is loss of lustre as observed visually. Decrease in density was also dinstinctly marked. But there is an increase in voids numbers with the rise of temperature. This is probably because of faster coagulation rate. At low temperature coagulation is retarded and more time is available for internal adjustment of polymer molecules leading to higher density.

Increase in coagulation temperature also brings about changes in cross sectionalship as apparent from microscopic study of proto fibre sample collected at $12^{o}C$, $16^{o}C$ and $20^{o}C$. At $20^{o}C$ the cross-section changed from kidney too slightly circular. The cross-sectional shape approaches a circular configuration, may be attributed to the greater infusion of water into the fibre causing it to be fluffy. The increase in voids are distinctly marked as the temperature goes up which is clearly seen in the photographs.

FINAL FIBRE

With the decrease in bath temperature, there is an increase in breaking tenacity and decrease in elongation. This may be due to increase in no. of voids. Final fibres were compared for rate of dyeing and dyeing depth. There is a marginal change in rate of dyeing with change in coagulation bath temperature but very slight change in depth was observed at higher temperature. This may be due to the aggregation of dyestuaff at surface in case of fibre with larger no. of voids. It is practically found that when the no. Of voids are more depth of shade is deeper but the fastness is lesser. At $20^{o}C$ bath temperature, very slight change in cross section was observed which was little bit towards circular. This has gor similar reason as in case of proto fibre.

Jet - Stretch

Eventhough jet stretch has lesser importance in acrylic fibres spinning an attempt was made to practically see the effect. A standard polymer composition was taken & fibres are prepared under standard spinning conditions. Only variable was jet – stretch. We observed that with increase in jet stretch from 1.06 to 1.10 there is decrease in fibre density & increase in no. Of voids as shown below.

TABLE- V FIBRE DENSITY

Sample	Jet stretch	Denier	Voids/C.S	Filament breakage
A	1.06	0.45	04	Normal
B	1.1	0.41	09	Higher

It has been physically also observed that increase in Jet stretch caused serious fibre rupture at take-up SS rollers. Also observed that wrapping tendency in stretching bath is higher. Therefore lower Jet stretch values are preferred in practical cases.

EFFECT OF STRETCHING CONDITIONS

To see the effect of stretching conditions, stretching ratio and stretching temperature were varied.

Stretching Ratio

For this study five different ratios were chosen, keeping all other conditions constant. Its effect on tenacity, Elongation, Degree of Crystallinity (%), orientation index (%) and filament breakage behaviour are assessed and shown in table- VI

TABLE-VI STRETCHING RATIO

Sample	S.R	Tenacity	Elongn.	Crystallinity (%)	Orientation index (%)	Filament breakages
A	5.5	3.2	40	51.2	32.8	Normal
B	5.8	3.4	38	59.4	39.2	Normal
C	6.0	3.6	35	63.2	43.9	A little higher
D	6.5	4.0	32	64.1	44.7	Quite higher
E	6.8	4.1	28	64.5	44.3	Quite higher

As seen from above table, while increasing stretch ratio from 5.5 to 6.5, a markable improvement was observed in tenacity but afterward only nominal increment was seen. The graph pattern is attached herewith also. As regard to elongation a marginal decreament was observed when SR was increased from 5.5 to 5.8 but afterwards the decrease was on higher side. Above phenomenon can be related to increase in both crystalinity and orientation index (%) till SR of 6.0 and while increasing SR from 6.0 to 6.5 almost a stabilization of oriented molecules occurred. Which caused sudden increase in tenacity from 3.6 gpd to 4.0 gpd. Then due to almost equivalency in the value of crystalinity and orientation index % the tenacity became stable.
As regard to filament breakage till the SR of 6.0. So it seems that the standard S.R should be around 6.0.

Stretching Bath Temperature

Stretching bath temperature influences the tenacity, elongation, DMF recovery and filament breakage a lot. So to have a vivid idea about this, test was carried out by taking three different set of temperature i.e., at 90^oC, 95^oC and 100^oC keeping all other conditions intact.

Its effect on various properties is studied and results are detailed below in table -VII

TABLE-VII STRETCHING BATH TEMPERATURE

Sample	Bath temp. (0^oC)	Tenacity	Elongn.	DMF Recovery	Crystalisation %	Filament Breakage
A	90	3.50	39	Quite low	68.2	High
B	95	3.20	38	Normal	66.1	Normal
C	100	2.41	32	Quite high	52.5	Quite high

The increase in bath temperature resulted in decrease in tenacity. This can be attributed to decrease in crystalinity %. It is to be noted that a higher temperature the drop in tenacity and crystalise % was considerably higher. The elongation did not show much of trend. The filament breakages was higher both at lower and higher temperature. From this point of view at the temperature of 90^oC even though the tenacity was high, the processing difficulties related to wrapping due to filament breakages was the barrier. The low DMF recovery at low temperature also is the probable cause for higher filament breakages. At higher temperature i.e., at 100^oC, the filaments were subjected to greater heat which caused maximum filament breakages. This indicates that there is an optimum temperature in terms of minimum filaments breakages which is in this case 95^oC.

EFFECT OF DRYING CONDITIONS

After coagulation and stretching, acrylic fibres tend to collapse in hot air drying technique. So it is imperative to study the behaviour of dried fibres after subjection of temperature profile and speed profile. In present study the temperature variants are taken for testing.

Temperature Profile

For the study samples of three trials were collected and evaluation of various physical properties specially, dyeing properties and other visual observations were noted and shown in table-VIII.

Apart from this other properties like tenacity, elongation, crystalline %, whiteness index, water retention are also tested.

Standard speed profile as taken for the study:

Drum	Speed (MPM)
Da	71.7
Db	71.0
Dc	65.0
Dd	59.4
D1	53.1
D2	45.6
D3	43.0
D4	42.6
D5	41.8
D6	43.7
D7	46.8
D8	50.0
D9	54.1
D10	53.4

TABLE-VIII EVALUATION OF VARIOUS PHYSICAL PROPERTIES SPECIALLY, DYEING PROPERTIES AND OTHER VISUAL OBSERVATIONS.

Profile	Void no.	Ten.	Elongn.	Crystallinity(%)	Orientation index(%)	Crimp stabillity(%)	Dyeing rate(%)	Dyeing depth	Whiteness index %	Water retention (%)
A	06	3.30	31.9	60.05	38.2	67	83	Slightly Darker	68	27.2
B	01-02	3.48	32.3	65.73	43.8	65	80	Darker	65	20.7
C	Nil	3.52	32.1	67.89	46.2	62	77	Normal	61	28.6

Profile	Zone-1	2	3	4	5	6	7	8
A	120	130	130	155	150	150	150	120
B	130	140	140	165	160	160	160	130
C	140	150	150	175	170	170	170	140

Dyeing Particulars

Temp - 90^{o}C
Time - 30 min
Blue dye - 1% Astrozen

If we analyse the above results, it is interesting to note that the voids numbers are coming down from 6 to 1 while increasing the temperature profile from “a” to “b” and to “0” at temperature profile “c”. It can be attributed to the collapsing of all the voids. The crystalline %, orientation index and tenacity increase with increase in dryer temperature profile.

As regard to crimp stability, it is just the reverse. A decreasing trend in crimp stability was observed while increasing temperature profile. It seems that at low temperature due to the presence of the moisture, it was on higher side, which modify the heat setting behavior. The dyeing rate decreased with increase in temperature. Dye depth does not show any particular trend. A decreasing whiteness index was observed with the increase in temperature profile. This is attributed to the dullness of the fibre at higher temperature. An interesting phenomenon of increase in water retention was marked while increasing temperature from "a" to "b" then comparatively less increase in water retention. This might be due to; at temperature profile "b" the voids are almost collapsed giving way to have maximum water retention. But after that due to marginal collapsing the water retention improved a little.

IV. SUMMARY AND CONCLUSION

Considering that use of acrylic fibre is extending to non-winter wear applications, tenacity of the fibre gains importance. From this context the present study relates to evaluation of acrylic manufacturing in terms of polymer and process conditions influencing tenacity value. It is found in present study, even though protofibre parameters influence the final tenacity value to a smaller extend, but the critical development of strength during spinning process is a must to withstand further drawing and drying. It has been found that the following proto fibre properties have positive influence on tenacity. Decrement of additives like MA increase the tenacity value. A value of 7.5 % MA gives a better result without disturbing other properties.

Increasing in dope concentration increase the tenacity and it was observed that a dope concentration of 25 % gives maximum tenacity with no other abnormality. Decrease in bath temperature increases the tenacity and a bath temperature of 12^oC gives better result. A part from this it was observed that bath composition and jet stretch do not play any role in influence tenacity. Considering the effect of stretching parameters it was found that stretch ratio has greater contribution for increase in tenacity. As observed the best result i.e. 4.0 gdp tenacity is achieved at 6.5 draw ration, but it gives rise to higher filament breakages causing wrapping. Therefore one has to compromise for a draw ration of 6.0 which gives a tenacity of 3.6 gdp.

Low bath temperature gives a better tenacity but it causes a higher filament breakage. At 90^oC the tenacity achieved is as high as 3.50 gdp. But it seems the optimum is 95^oC which gives a desired tenacity and normal filament breakage. It can be concluded that for increasing tenacity a draw ratio of 6.0 can give better result than decrease bath temperature upto 90^oC.

As regards to dryer parameters it is observed that the temperature profile does not influence the tenacity much. From the processing point of view the constrains of time and physibility of changes made the study restrictive. The study confined to only particular denier of fibre. Hence a comprehensive study would be necessary to confirm the commercial applicability of the present findings.

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