EFFECT OF ALKALI PRETREATMENT AND DYEING ON FIBRILLATION PROPERTIES OF LYOCELL FIBER

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Abstract : Lyocell is a new generic name given to a cellulosic fiber which is produced under an environmentally friendly process by dissolving cellulose in the tertiary amine oxide N-methylmorpholine-N-oxide (NMMO). Lyocell fiber shows some key advantageous characteristics over other cellulosic fibers; for instance, a *high dry and wet tenacity and high wet modulus*, but one disadvantage of this fiber is generating of fibrillation during the wet state, which causes the formation of longer and more oriented crystalline regions and smaller but more oriented amorphous regions in the fiber structure. But it has been proven to be *disadvantageous for some other applications*, such as the launderability of the product and difficulty to control the uniformity of color uptake during dyeing, and Pills formation and streak marks in dyeing.

In this paper dealt with the effect of alkali and dyeing treatment of lyocell on fibrillation properties.
Key words: Alkali treatments, Defibrillation, Fibrillation tendency, Polyfunctional reactive dyes.

1.1 Introduction

Highly oriented cellulosic fibers such as cotton, cuprammonium and polynosic rayon, fibrillation of lyocell is greater. Fibrillation is the *longitudinal splitting of a single fiber* into microfibers of typically less than 1–4 μm in diameter. In the swollen state lyocell has an extensive fibrillation tendency owing to linear high crystalline fibrillar morphology (1, 2). It is one of the important properties of Lyocell. Due to the unique highly crystalline structure of lyocell, and weaker lateral links between the crystallites, the fibers undergo localized separation of fibrous elements at the surface known as fibrillation, mainly under conditions of wet abrasion (3,4,5,6). The fibrils formed can be so fine that they become virtually transparent and give a frosty appearance to the finished fabric. If fibrillation is not controlled, these microfibers become entangled giving a serious problem of ‘pilling’. It also weakens the mother fiber; also appearance of fabric is become totally unacceptable. It is well known that the fibrillation tendency of Lyocell fibers is related to swelling state. In view of this, it is necessary to examine the effect of different types of alkali (Sodium hydroxide (Na OH), Lithium hydroxide (Li OH), Potassium hydroxide (K OH), Tetra methyl- ammonium hydroxide (Tm AH) at room temperature on Lyocell fibers (8,9,10,11,12,13,14,15). Specific multifunctional reactive dyes are reported to have favorable effect on fibrillation behavior of Lyocell fiber. The cross linking of reactive groups of these dyes with adjacent cellulose chains provides an opportunity to reduce fibrillation during wet processing (16,17,18,19,20,21,22,23).

2. Experimental Procedure

2.1 Materials

Knitted Lyocell fabrics, Lyocell fibers supplied by Lenzing AG were used for experiments. The geometrical properties of the fabric are given in Table-1.

Table 1: *Particulars of Lyocell Fabric*

Properties	Course/ Wales Count (Ne)	CPI & WPI (Inch)	Weight (grams)	C.S.P
Descriptions	24 / 24	36 and 56	150	3360

2.2 Dyes & chemicals

Lithium hydroxide, sodium hydroxide, potassium hydroxide and tetramethylammonium hydroxide (TMAH, 25% sol.), Polyfunctional Reactive dyes (C.I Red 286) were AR grade and used without further purification.

2.3 Pretreatment

Lyocell fiber was soaked into alkali solutions of certain concentration for 30 minutes at room temperature, after neutralization with an acetate buffer solution (pH=5) and rinse with hot and cold water, it was dried at 60 °C

***Table 2**: Process conditions for pretreatment*

Samples	Alkali Name	Sample Code	Concentration (g/l)	Temp °C
1	Na OH	N1	1	Room Temperature
2		N2	2	
3		N3	3	
4	KOH	K1	1	
5		K2	1.5	
6		K3	2	
7	Li OH	L1	0.5	
8		L2	1	
9		L3	1.5	
10	Ta OH	T1	0.25	
11		T2	0.5	
12		T3	1	

2.4 Dyeing

The alkali treated fabric was dyed with polyfunctional reactive dyes. Dyeing of fabric carried out in two methods, process conditions shown in figure:1,

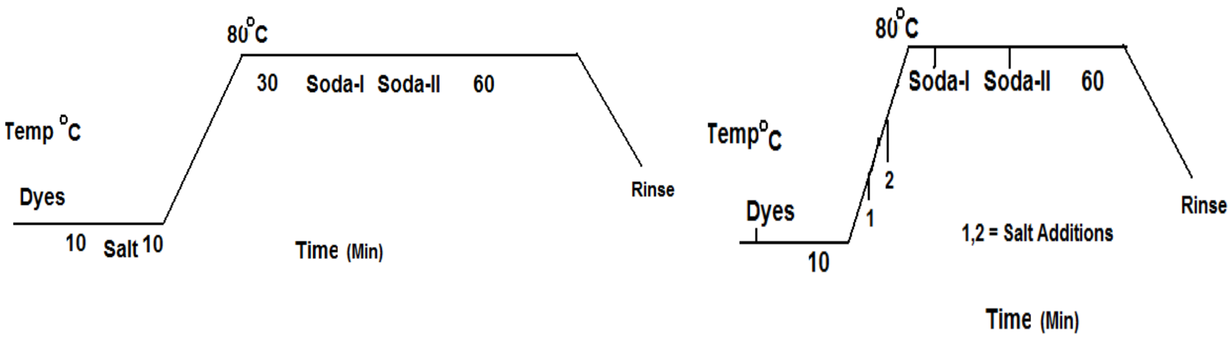

***Figure 1:** Dyeing Temperature Profile (HE & Migration Method)*

***Table 3:** Process conditions for dyeing(HE and Migration Method)*

Shade (%)	1
Dyeing Temperature	80 °C
Na_2CO_3	8-11 gpl
NaCl	15 gpl
pH	10-11
Time of Dyeing	60 min

2.5 Test Methods (24,25,26,27)

The physical and chemical properties of the alkali treated, dyed fabric samples and the instruments used are given in Table 4.

***Table 4:** Physical properties of Lyocell fabric sample*

S.no	Property	Standards	Instrument used
1.	Wash fastness	*AATCC-107/2002*	Wash fastness tester (Landerometer)
2.	Rubbing fastness	*AATCC-008/2005*	Crock meter
3.	Pilling Resistance	ISO 12945-1	ICI Pill box tester
4.	Abrasion Resistance	ISO 12947-2	Martindale abrasion tester

3. Result and Discussion

3.1 Effect of Pilling Resistance on Alkali treated and Polyfunctional reactive dyed (H.E & Migration) lyocell

The Lyocell fabric laundered ten times as per the AATCC 135-2004 standard, then observes the pilling resistance as per the ISO 12945-1 grade. Typically pilling grade is excellent when the concentration of alkalis is increase, as well as the type of alkali used, also the same fabric were dyed with PF reactive dyes means it show the excellent pilling resistance, As per the ISO 12945-1 standard, the grade 5 represent the outstanding pilling resistance, as well as 1 represent the very poor pilling resistance. As per the one way Anova, various pretreatment is not significant on pilling resistance, in case of same pretreated sample were dyed with PFRD shows significant on pilling resistance.

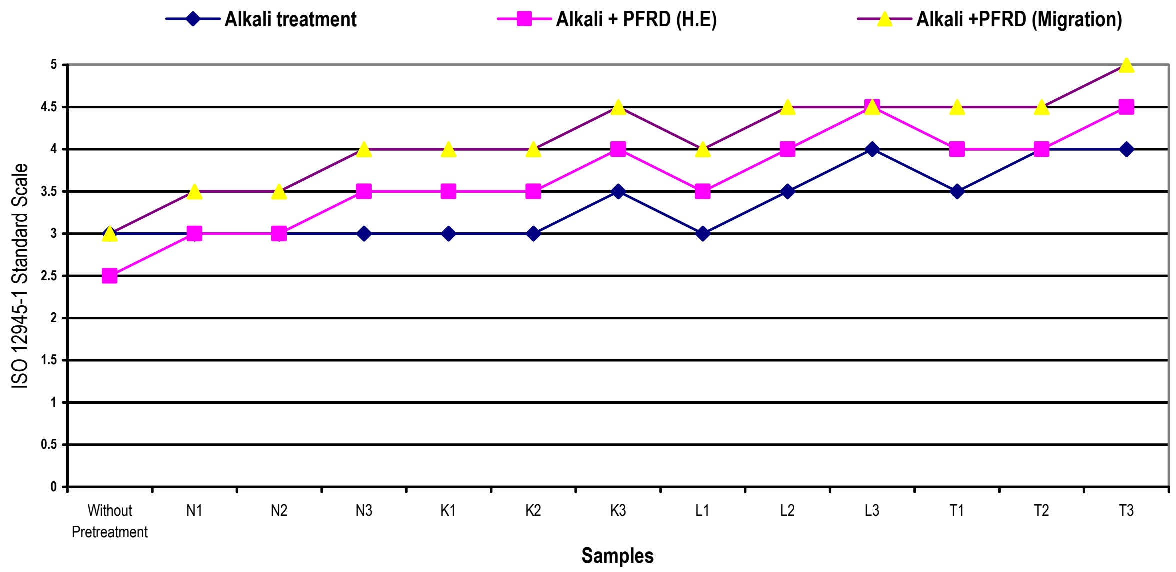

***Figure 2:** The graphical representation of the pilling test on Alkali treated and Polyfunctional reactive dyed (H.E & Migration) lyocell*

3.2 Effect of Abrasion Resistance Alkali treated and Polyfunctional reactive dyed (H.E & Migration) lyocell

The Lyocell fabric laundered ten times as per the AATCC 135-2004 standard, then observes the abrasion resistance determined with a Martindale abrasion resistance tester according to ISO 12947-2- 1999 method. *For this present study*, both parameters such as concentration and type of alkalis are directly inclined the abrasion resistance of lyocell fabrics, typically Abrasion resistance was excellent when the concentration of alkalis is increase, also the same pretreated fabric were dyed with PF reactive dyes show the excellent abrasion resistance, as per the one way anova various pretreatment and dyeing with PFRD is significant on abrasion resistance. *Figure 3 Abrasion Resistance test results on Alkali treated and Polyfunctional reactive dyed (H.E & Migration) lyocell.*

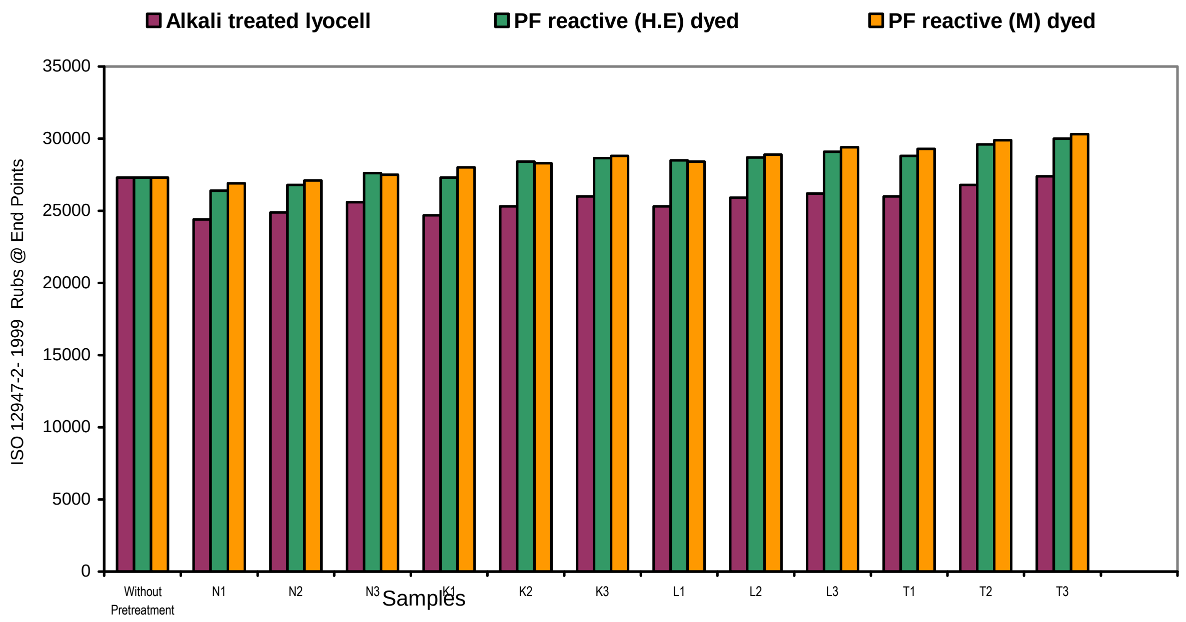

Figure 3: *The Bar chart representation of the Abrasion resistance test on Alkali treated and Polyfunctional reactive dyed (H.E & Migration) lyocell*

3.3 Effect of Color Strength on Dyed Lyocell

- The test results were analyzed with the standard of AATCC 182: 2005
- K/S value Conventional dyed sample is comparatively lower than the K/S value of Polyfunctional Reactive dyed lyocell.
- K/S value is found to be more than High Exhaustion method when same concentration used in migration method.
- Maximum K/S value found at combination of 1 gpl of Tetra methyl- ammonium hydroxide and followed by Polyfunctional reactive (Migration) dyeing.

3.4 SEM Analysis on Alkali Treated and Polyfunctional Reactive Dyed (H.E & Migration) Lyocell

In order to clarify the structural change during alkaline treatments, image analysis was performed using *SEM*. Figure 4 to 10 gives the images of surface structure of fiber treated in different alkali solutions. The large bundles or layers of macro fibrils are clearly observed on the surface of the fibers treated with different alkali solutions. The fiber treated with 1 gpl of Tetra methyl- ammonium hydroxide shows a smooth surface without any bundle and layer of macro fibrils, the same samples were dyed with PolyFunctional

Reactive Dyes (PFRD), (particularly migration methods) shows excellent smooth surface without any bundle and layer of macro fibrils (fibrillation),

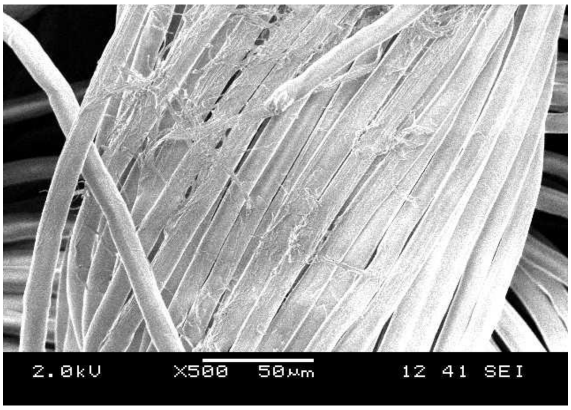

Figure 4: *Scanning Electron Microscopic picture of original Lyocell fiber*

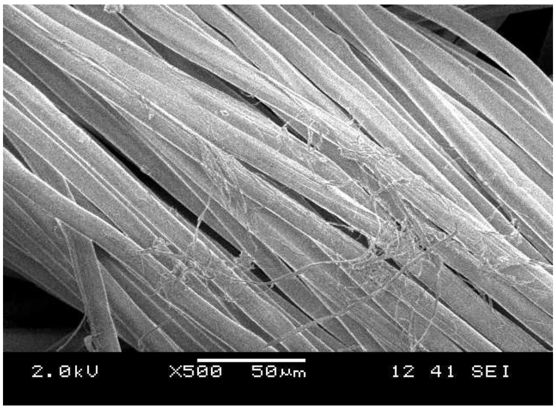

Figure 5: *Sodium hydroxide treated Lyocell fiber*

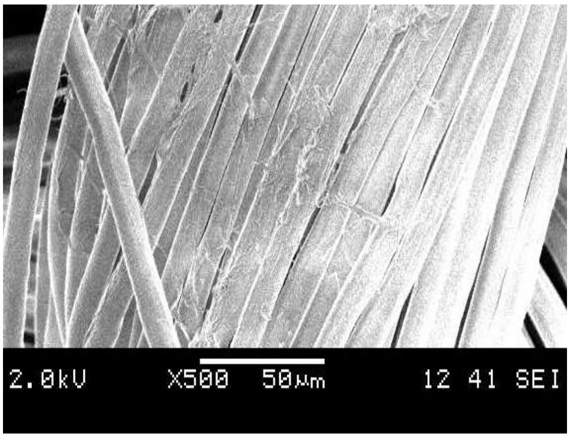

Figure 6: *Potassium hydroxide treated Lyocell fiber*

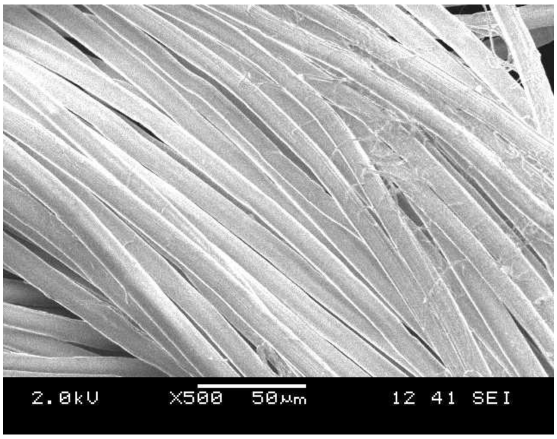

Figure 7: *Lithium hydroxide treated Lyocell fiber*

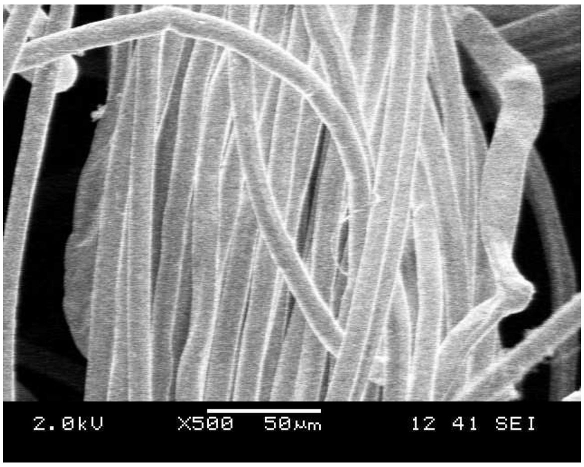

Figure 8: *Tetra methyl- ammonium hydroxide treated Lyocell fiber*

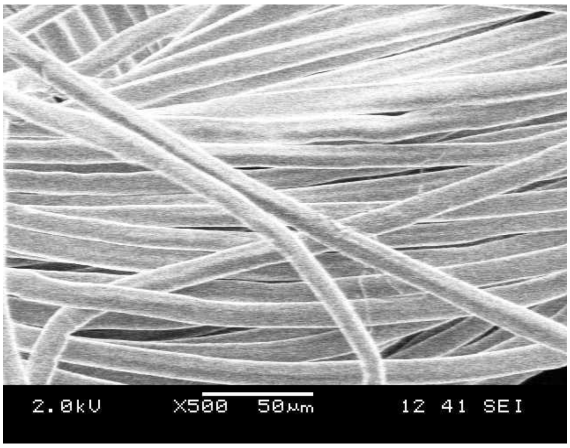

Figure 9: *Alkali treated and PF Reactive Dyed (H.E) Lyocell fiber*

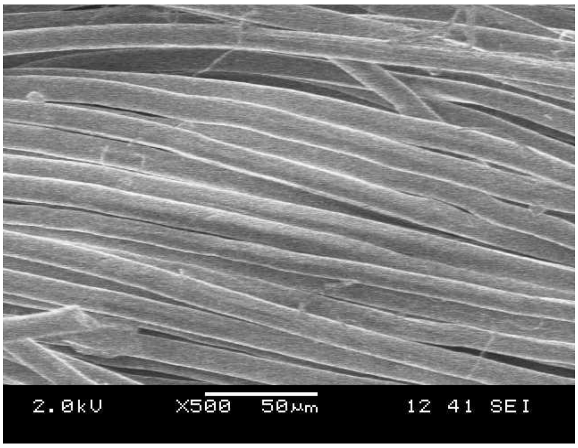

Figure 10: *Alkali treated and PF Reactive Dyed (migration) Lyocell*

4 .1 Conclusion

- Pretreatment of the lyocell fabric with various alkalis and followed by dyeing with polyfunctional reactive dyes resulted in mark reduction in fibrillation.
- The above pretreatment and dyeing resulted in improved pilling resistance and abrasion resistance.
- Due to excellent exhaustion and fixation of polyfunctional reactive dyestuff (95%) leading to more cross linking with hydroxyl group of lyocell, and it cause to reduce the fibrillation formation and further improvement of abrasion resistance and pilling resistance, also it produce excellent wash and rubbing fastness.
- Scanning Electron Microscope images shows, TmAH treated Lyocell have excellent smooth surface and without any bundle layers of macro fibrils.
- The above pretreated sample (1 g/l TmAH) when dyed with polyfunctional reactive dyes shows further improvement of surface appearance without any macro fibrils.

4.2 Reference

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APPENDIX

***Table :6** Abrasion Resistance test results on Alkali treated and Polyfunctional reactive dyed (H.E & Migration) lyocell*

S. no	Sample code	Abrasion Resistance value Pressure 9 Kpa (End Point Found @ Rubs)		
		Alkali treated	Dyed with PFR (H.E)	Dyed with PFR (M)
1.	Without Pretreatment	27300	27300	27300
2.	N1	24400	26400	26900
3.	N2	24900	26800	27100
4.	N3	25600	27600	27500
5.	K1	24700	27300	28000
6.	K2	25300	28400	28300
7.	K3	26000	28650	28800
8.	L1	25300	28500	28400
9.	L2	25900	28700	28900
10.	L3	26200	29100	29400
11.	T1	26000	28800	29300
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
13.	T3	27400	30000	30300

***Table :5** Pilling Resistance test results on Alkali treated and Polyfunctional reactive dyed (H.E & Migration) lyocell*

S. no	Sample code	Pilling Resistance value		
		Pre Treated	Dyed with PFR (H.E)	Dyed with PFR (M)
1.	Without Pretreatment	3	2-3	3
2.	N1	3	3	3-4
3.	N2	3	3	3-4
4.	N3	3	3-4	4
5.	K1	3	3-4	4
6.	K2	3	3-4	4
7.	K3	3-4	4	4-5
8.	L1	3	3-4	4
9.	L2	3-4	4	4-5
10.	L3	4	4-5	4-5
11.	T1	3-4	4	4-5
12.	T2	4	4	4-5
13.	T3	4	4.5	5

***Table : 7** Effect of color strength on Alkali pretreated and Dyed with PFRD (H.E & Migration) lyocell*

S.no	High Exhaustion Method		Migration Method	
	Sample Code	K/S Value	Sample Code	K/S Value
1.	Without Pretreatment	10.7	Without Pretreatment	10.7
2.	N1	11.2	N1	11.4
3.	N2	11.6	N2	11.8
4.	N3	11.9	N3	12.3
5.	K1	11.2	K1	11.3
6.	K2	12.0	K2	12.1
7.	K3	12.3	K3	12.8
8.	L1	11.6	L1	11.9

9.	L2	12.3	L2	12.7
10.	L3	12.9	L3	13.4
11.	T1	11.9	T1	12.4
12.	T2	12.7	T2	13.2
13.	T3	13.0	T3	13.9

Table : 8 *Color and Rubbing Fastness test results of Pretreated with alkalis and dyed with PFRD (High Exhaustion method)*

Sample No.	Sample code	Colour fastness to washing		Colour fastness to rubbing	
		Shade change	Staining on cotton	Dry rub	Wet rub
1	N1	4	4	3	3-4
2	N2	4	4	3-4	3-4
3	N3	4	4	4	3-4
4	K1	4	4	3-4	4
5	K2	4	4	4	3-4
6	K3	4	4	4	4
7	L1	3-4	3	4	3-4
8	L2	4	3-4	3-4	3-4
9	L3	3-4	3	4	3-4
10	T1	4	4	4	4
11	T2	4	4	4	4
12	T3	4	4	4	4

Table : 9 *Color and Rubbing Fastness test results of Pretreated with alkalis and dyed with PFRD (Migration method)*

Sample No.	Sample code	Colour fastness to washing		Colour fastness to rubbing	
		Shade change	staining on cotton	Dry rub	Wet rub
1.	N1	3-4	4	4	3-4
2.	N2	4	4	4	4
3.	N3	4	4	4-5	4
4.	K1	3-4	4	4	4
5.	K2	4	4	4	4
6.	K3	4	3-4	4-5	4-5
7.	L1	3-4	3-4	4	3
8.	L2	4	3-4	4	3
9.	L3	4	4	4-5	4
10.	T1	4	4	4	4
11.	T2	4	4	4-5	4
12.	T3	4-5	4	4-5	4-5