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Impact of Collapsible Tube's Lacquer Coating and Its Porosity Due to Printing and Fabrication of Tube

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ABSTRACT

The Idea of Study is to check the effectiveness and impact of lacquer coating quality after post fabrication and printing process. The porosity test is used to check the uniformity of lacquer in collapsible tube, after internal lacquering there are various methods left to make final printed tube (ref flow chart 1). During these processes the collapsible tube may get minor damage that may damage the uniformity of internal lacquer coating. Since uniform lacquer coating is very important for product compatibility and quality for long term study and use. Hence this study has done on printed and unprinted lacquered tube to see the porosity uniformity of lacquer. After observation, data study (Nos. of dark spot deposition) we can not found any major difference in dark spot generation (porosity test) for both type of tube. Also we kept one drug product in both printed and unprinted tube after two month study there is no significance difference observed during stability.

Keywords: Collapsible tube, Lacquer coating, Impact extrusion process, porosity test, Electrolytic Deposition, Cupper deposition.

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INTRODUCTION

Collapsible tubes made of tin, tin alloy or aluminium as they are majority of medical creams and ointments are marketed in collapsible tubes made from aluminium[9]. Metal tubes are impermeable to moisture, gas, and odour and light provided they are adequately closed[1]. Aluminum has superior "dead fold" characteristics, which give it the ability to remain bent or folded without rupturing. This quality is necessary for a container that is designed to be squeezed and folded as the dispensing method[11].

They are convenient for a customer or patient to use and as the contents are expelled by squeezing the tube, there is no tendency for the walls to recover their original shape when the pressure is released. Consequently the risk of air entering the pack and reacting with the product or causing it to dry out is minimized. Internal coating may be necessary to prevent chemical reaction[1]. The tube itself is absolutely scent-neutral and can be sterilized and coated with an inert internal lacquer. Aluminium permanently prevents the penetration of oxygen. This essential barrier property makes the aluminium tube a most appropriate and well established packaging in the food, cosmetic and pharmaceutical markets[8]. The ductile metals used for collapsible tubes are tin (15%), aluminum (60%), and lead (25%). Tin is the more expensive than lead. Tin is the most ductile of these metals. Laminates of tin-coated lead provide better appearance and will be resistant to oxidation. They are also cheaper compared to tin alone. The tin that is used for this purpose is alloyed with about 0.5% copper for stiffening. When lead is used, about 3% antimony is added to increase hardness[8].

Manufacturing process:

The impact extrusion process is used to produce open-ended collapsible tubes (Figure 1.0).From softer metals such as tin and lead. When aluminium is used, it work-hardens during the forming process and the resultant tubes must be annealed to regain flexibility. Alternatively aluminium tubes may be left in their work-hardened state as rigid containers. Impact extrusion is a particularly useful process to produce containers with a high length to diameter ratio, e.g. up to 7:1[2]. The formed tubes then pass to a trimming machine where they are cut to length, a thread cut or rolled on the nozzle, and the face of the nozzle orifice cleaned. The shoulder, which is relatively rigid, may be decorated, e.g. with concentric rings, if required. The tubes are then ready for the finishing operations of internal coating, enamelling, printing and capping[1]. When internally lined with the appropriate lacquer, collapsible aluminium tubes serve as a stable corrosion-free packaging[9].Aluminium tubes must be annealed after forming and finishing, otherwise they are too springy this process also serves to remove all traces of lubricant. Tin is the least reactive of the metals available, is very bright and is also non-toxic. However, it is inherently expensive and its usage is therefore restricted to pharmaceuticals such as antibiotic and some ophthalmic ointments where maximum protection is required. Lead-based tubes are now not recommended for pharmaceutical products, for toxicity reasons[9]

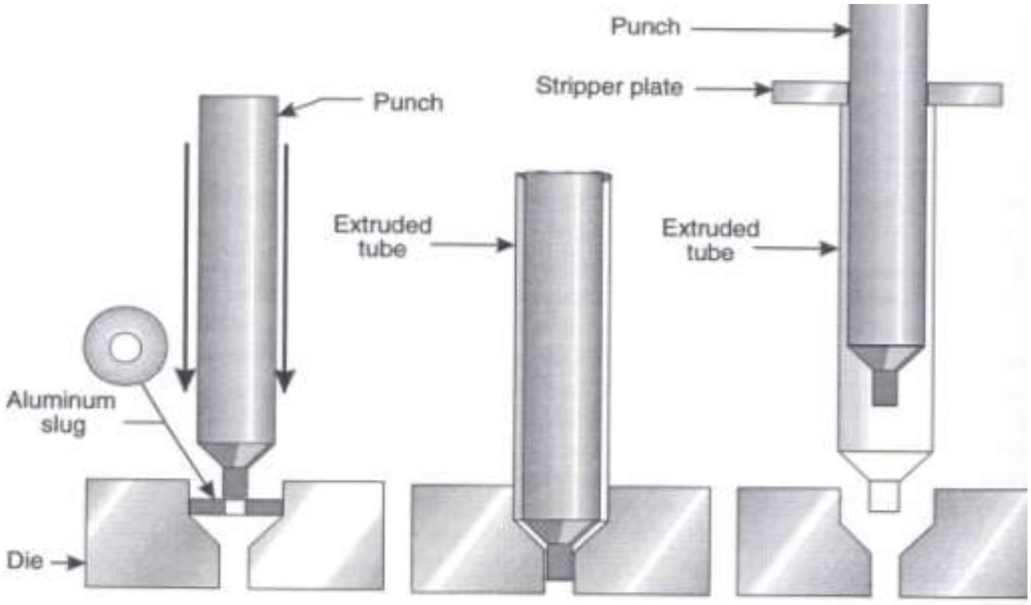


Figure 1.Collapsible metal tubes produced by impact extrusion[1]

USE OF COATINGS FOR COLLAPSIBLE PACKAGING

A wide range of coatings are used including vinyl, epoxy and phenolic resins. The use of epoxy and phenolic resins is restricted to aluminium tubes due to the high curing temperatures required. Most internal lacquering systems involve two coatings, the first being partially dried before applying a second coat and drying completely. Needless to say, where lacquers are used it is essential that they must be pinhole-free for the whole length of the inside of the tube, including the interior of the nozzle. After the internal coating has been applied the tubes are enamelled externally, baked, printed and the print dried by heating again[9].

Collapsible tube packaging is coated for many reasons such as:

Internal (product contact) coatings[4]:

• Provide protection of the contents from the metal.

•Provide protection of the tube from the contents of the tube – e.g. acidic, alkaline or sulphur product may couse problem.

External (non-product contact) coatings[4]:

• Provide protection of the metal from the environment – e.g. atmospheric corrosion

• Support decoration, labelling and consumer information

• Influence mobility (friction) of the article during filling operations

• By reducing tool wear for tin-free steel (TFS) substrates. Tin on the surface of steel (electrolytic tinplate – ETP) "lubricates" the metal during deformation, whereas steel without a tin layer is very abrasive and the presses used for forming would rapidly wear out

• By assisting tooling for aluminium substrates.

Coatings are applied to metal and after thermal treatment (cure schedule or staving) form a dry (final) film on the metal. Most coatings are applied as a wet film. The major constituents in a coating as applied to the metal include:

• Resin(s)

• Cross-linking agents (almost always present)

• Additives

• Solvents (not always present).

Table 1: Types and properties of resins used in internal can coatings[4]

Type	**Nature**	**Flexibility**	**Pack resistance**	**Main end-uses**
Epoxy-phenolic	High molecular weight epoxy resins cross-linked with phenolic resole resins	Good	Very good	Universal gold lacquer for three-piece cans Shallow drawn cans
Organosol	PVC dispersed in an appropriate varnish and conventionally stabilised with a low molecular weight epoxy resin or novolac epoxy resin. Epoxidised oils can also be used	Very good	Very good	Drawn cans Easy-open ends Often used over epoxy phenolic basecoat.
Epoxy-anhydride	High molecular weight epoxy resins cross-linked with anhydride hardeners	Good	Very good	Internal white for three-piece cans
Epoxy-amino	High molecular weight epoxy resins cross-linked with amino resins. Also Side seam stripes epoxy acrylic water-Some food systems based spray internals for B&B DWI	Good	Limited	Universal lacquer for beer and beverage cans (water reducible)
Polyester	Polyester resins cross-linked with Resins. May contain lower Molecular weight Epoxy resin	Very good	Pack - dependent	May not be suitable for very acidic and amino or phenolic aggressive foods

The first three components are incorporated into the dry (final) film. In case a solvent is used, it evaporates during the cure schedule. The film, which is in contact with food, must comply with relevant food regulations as discussed in the chapter on regulatory aspects Unlike most plastics, the majority of coatings only attain their final properties after the wet (applied) film has undergone further chemical reactions, normally during the cure schedule. Typically the resin(s) would react with one or more cross-linking agents (or resins), which join individual resin molecules together to form a three-dimensional cross-linked network. It is this network and the density of crosslinks in combination with the different molecules used in the resins that give the corrosion resistance and flexibility, amongst other properties, of the final film[4].

Lacquer damage[5]:

Three main types of damage are common with impact extrusions, particularly with collapsible tubes, all of which substantially impair the corrosion resistance of the aluminium itself, and the lacquer films.

These are as follows:

1. Excessive extrusions Marks[5]:
2. Cross scoring[5]:
3. Knurl marks[5]:

There are three main causes of loss of adhesion in use, assuming at the coating material is not at fault in this respect, and that optimum staving conditions have been observed[5]:

1. Lubricant Residues remaining under the dried lacquer film:
2. Film Penetrators in the product:
3. Oxide films on the metal:

Printing process of collapsible tube rotary dry offset letterpress:

Metal containers produced by impact extrusion must be decorated as the last stage rather than the first stage. The process normally used is rotary dry offset letterpress[2]. This is a relief printing process in which the image areas are raised above the level of the non-image areas and transferred from the inked printing plate to the metal surface by means of an intermediate resilient rubber surface[1]. A dry offset process using either thermally or UV-cured inks. The tube is pushed on a mandrel and rolled past a curved printing blanket, which applies up to six colors simultaneously[2]. A base coat of white enamel is first applied to the container by roller coating and set by partial baking, which aids keying of the inks at the printing stage. The base coat can also extend marginally around a corner radius, e.g. at the base of a rigid tube or over a bead or shoulder where present. However, the next operation of printing itself is restricted to the cylindrical surface as the actual (relief) printing plate is wrapped around a cylinder in a similar manner to a lithographic plate. Each tube is supported separately on a mandrel which can rotate freely on its axis. The inking stations apply their separate images to the same rubber-faced blanket cylinder and the composite image is transferred to the tube in a single revolution of the latter. The printed tubes are then dried again. The tubes are held on pins for both the initial base coat baking cycle and the print baking cycle. A period of 4 min at 170–230°C is normally adequate, depending on the nature of the enamel & the inks. Where there is a possibility of the product reacting with decoration, an over-varnish is applied by roller coating & set by baking[1].

MATERIALS AND METHOD

Electrolytic Deposition of Copper Method:

The pores in the lacquer layer are detected by electrolytic method with the help of direct current of a certain voltage and ampere. Spongy copper is deposited on the pores from the copper sulphate solution.

Test Solution:

The solution of following composition shall be used for the test:

Copper Sulphate ($CuSO_4 . 5 H_2O$)	5.0 parts (m⁄v)
Glacial acetic acid	0.5 parts (m/v)
Distilled water	94.5 parts (m/v)

Source of Current:

Direct current of 6 volts and a maximum of 2.5 amperes from a battery charger shall be used.

Procedure:

Split opens the tube and flattens it. Bend up about 1 cm of each of the four sides and squeeze the corners together in such a manner that an internally lacquered through is formed. The size of the trough should be constant for all tubes of the same size. Fill the trough with the test solution. Connect the negative pole of the current with the aluminium side of the tube. Bend a copper wire approximately 4 mm in diameter so as to run parallel to the lacquer surface and extend over at least half the length of the trough. Attach the wire to the positive pole and immerse into the test solution for 10 seconds.

Figure 1: Solutions, tube sample and battery for experiment

A dark spot deposit of copper is formed at porous points. To evaluate the porosity in the neck and shoulder region, screw the cap on and cut the tube round up to a height of about 2 cm above the shoulder, leaving a small projection to connect the negative pole. Fill the tube with solution and dip the copper wire inside almost to the nozzle.

Figure 2: Experiment performance

Requirements:

If no copper is deposited, the tube is entirely free from pores. The maximum number of porosity deposits for each diameter of tube shall be as given below:

Table 2: The maximum number of porosity deposition (dark spot) limit for different diameter of tube

Tube diameter, mm	Maximum number of porosity deposits of size 0.5mm diameter and above
13.5	5
16	10
19	10
22	15
25	15
30	20
35	20

Observation:

1) Black dark spot was observed when we apply current.

2) When the time of current flow was increasing, gradually nos. of dark spot increased; also the diameter of dark spot was increased.

3) The probability of dark spot generation was higher around previous generated spot; also other dark spot was generated just adjacent to dark spot.

4) The experiment was performed on both printed and unprinted tube, there was no significant difference observed on both type of tube.

5) After certain time period the dark spot generation gets stopped, but the diameter of dark spot gets increased till exposed metal layer was present.

6) On the basis of stability study, we observed that there is no variation in stability data result for both printed and unprinted tube.

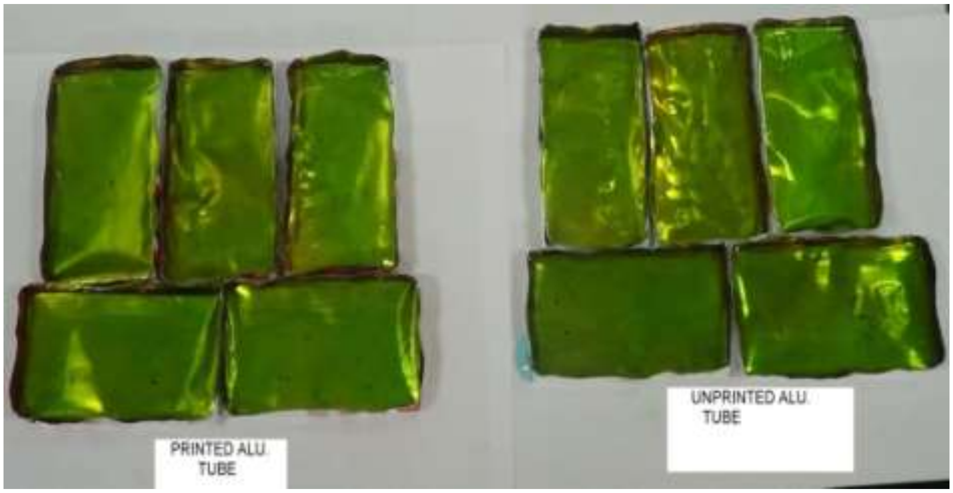


Figure 3: Observation of black dark spot after 10 sec on printed and unprinted alu tube

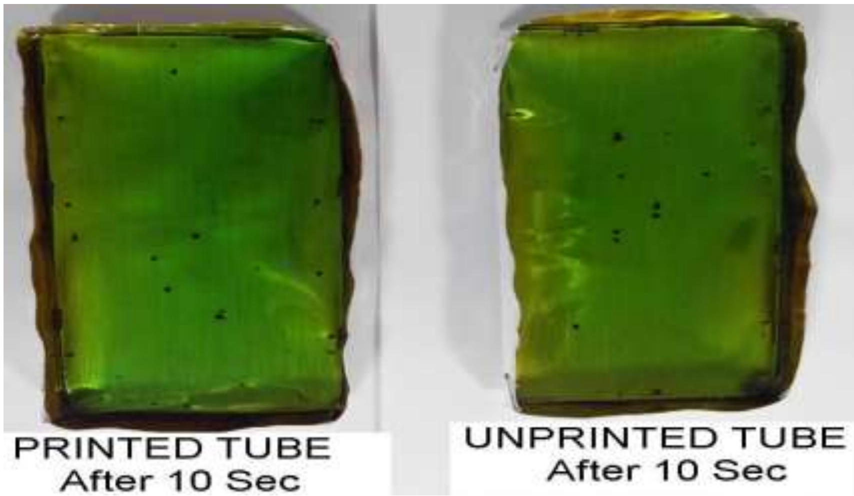


Figure 4: Observation of black dark spot after 10 sec on printed and unprinted alu tube

Figure 5: Observation of black dark spot after 20 sec on printed and unprinted alu tube

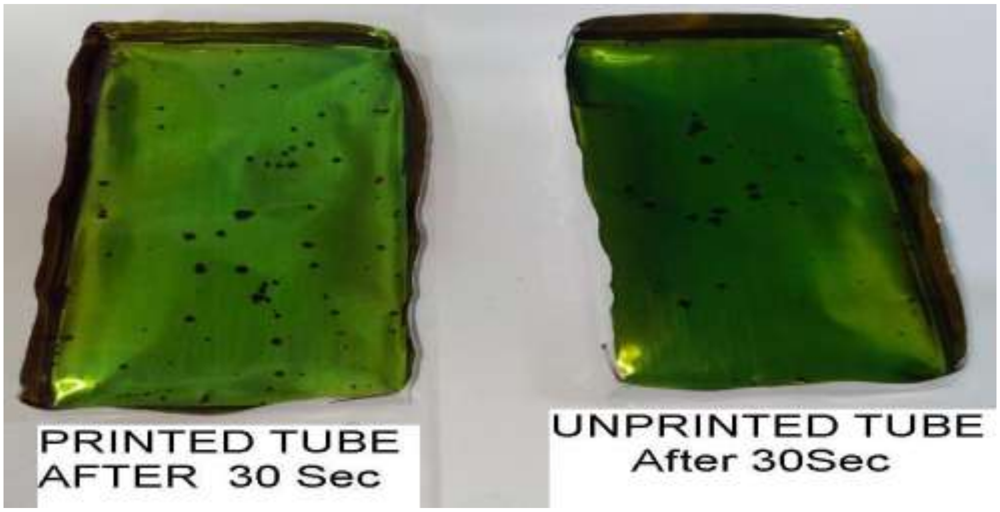


Figure 6: Observation of black dark spot after 30 sec on printed and unprinted alu tube

RESULTS AND DISCUSSION:

The generation of dark spot shows there were exposed metal area i.e. without lacquer area were present on inner layer of tube, that cause chemical reaction to form dark spot. As the time of exposure of current increased, causes increased in chemical reaction, hence the deposition of dark spot gets enlarges, also new spot created near other dark spot since more tempering, wear and tear was taken place at that region

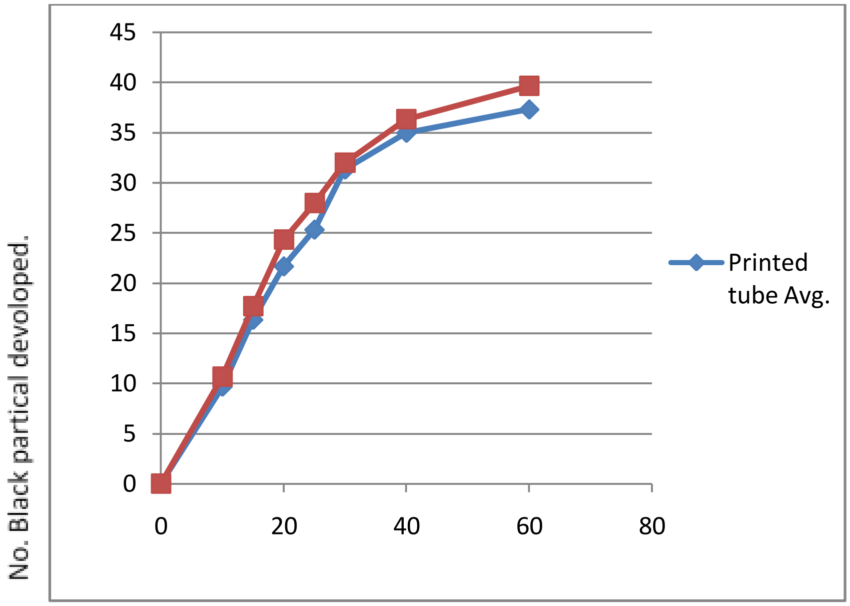
Table 3: Avs. nos. of black dark spot developed Vs Time (sec) for printed and unprinted alu tube

Sr. no	Alu tube & nozzle	After 10 sec. No. of black dark spot develop	After 10 sec. No. of black dark spot develop	After 15 sec. No. of black dark spot develop	After 20 sec. No. of black dark spot develop	After 25 sec. No. of black dark spot develop	After 30 sec. No. of black dark spot develop	After 40 sec. No. of black dark spot develop	After 60 sec. No. of black dark spot develop
		-	A	B	C	D	E	F	G
1	Printed tube(P-1)	0	6	8	13	14	24	27	29
2	Printed tube(P-2)	0	11	24	24	30	34	40	43
3	Printed tube(P-3)	0	12	17	28	32	36	38	40
Appx. Avg		0 0	10	16	22	25	31	35	37
4	Unprinted tube(UP-1)	0	11	19	28	32	37	40	48
5	Unprinted tube(UP-2)	0	12	16	23	27	32	38	33
6	Unprinted tube(UP-3)	0	9	18	22	25	27	31	38
Appx. Avg		0	11	18	24	28	32	36	40

PSI- Partical size increases

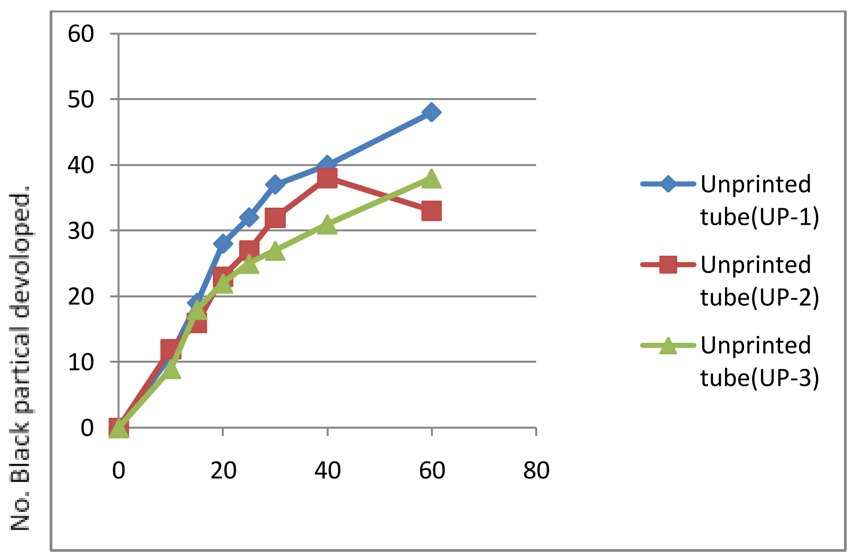
Conclusion of table:

During study we observe that there should not be major difference in black dark spot size printed & unprinted alu tube.



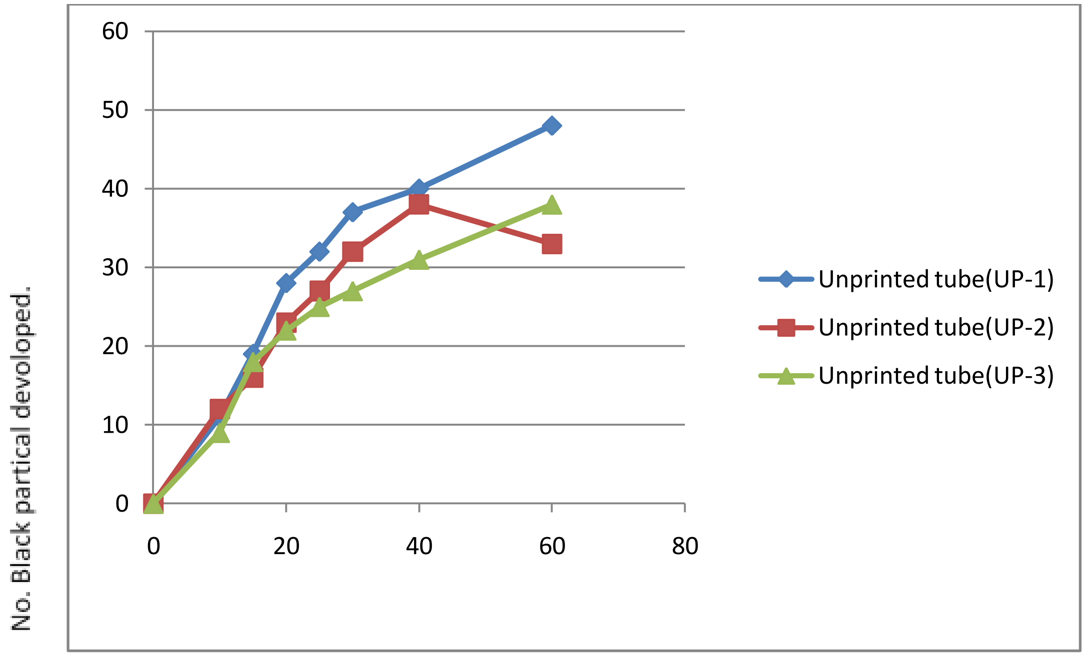
Time in Sec

Graph -1: Avs. Nos. of black dark spot developed Vs Time (sec) for printed and unprinted tube



Time in Sec

Graph -2: Avs. Nos. of black dark spot developed Vs Time (sec) for printed tube



Time in Sec

Graph -3: Avs. Nos. of black dark spot developed Vs Time (sec) for unprinted tube

CONCLUSION:

On the basis of pattern of dark spot generation shows that there is no much significant difference occurs for printed and unprinted tube hence we can conclude that there is not much difference occurs on lacquer quality during fabrication and printing process also the product stability data shows similar result on both printed and unprinted tube.

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