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COMPARATIVE STUDY ON FILTER EFFICIENCY OF DIFFERENT BRANDS OF CIGARETTES BY MICROSCOPIC AND SPECTROSCOPIC TECHNIQUES

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ABSTRACT: Internationally there has been research on large scale for determination of efficiency of cigarette filters but in India it still remains a topic of discussion. Being the second largest consumers of tobacco and tobacco products and one of the largest and fastest growing consumers of cigarettes, such studies need to be undertaken at a greater level to assess the risk and hazards of cigarette smoking. The government of India seems to focus more on the taxation policy on tobacco products with a view that increased taxes on tobacco products will make it difficult for common masses to consume them. However, the Global Adult Survey conducted by the Government of India, Ministry of Health and Family Welfare clearly indicates that despite of increased taxes on tobacco and tobacco products, the number of consumers have grown many folds in last one decade. Most of the researches have used smoking machine regime for determination of human smoke yields from spent filter, however as per the WHO, no smoking machine can actually mimic the normal human smoking behaviour. Hence, keeping this aspect in mind, the current research is focused on estimation of filtration efficiency from spent cigarette filter without the use of smoking machine. Though there are numerous factors that can cause variation in filtration efficiency of cigarette filters but irrespective of these aspects this research project was selected for the comparative study on filter efficiency of different brands of cigarettes available in India by microscopic and spectroscopic techniques.

INTRODUCTION: The analysis of spent filters from human-smoked (HS) cigarettes has been used for last three decades for estimation of cigarette yields. It is considered as one of the least invasive methods of estimating HS cigarette yields[1, 2]. There is a considerable variation in filtration efficiency that varies in a non-linear fashion according to the velocity of the smoke passing through the filter and some extent, the length of the tobacco rod smoked.

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The filtration efficiency increases significantly as the velocity of smoke through the filter (flow rate/cross sectional area) falls below 30 cm/s, but becomes relatively constant at higher velocities. In another key research, there was an attempt to minimize the flow-dependency effect by measuring nicotine FEs over a range of machine smoking regimes and using the average FE to determine nicotine yields from the filters collected from smokers(whole-filter method) [3].

The machine smoking used for analysis needs to be done on multiple days to incorporate day-to-day variability so that a calibration is achieved with that of yields from machine smoking. In addition, the tip extractions and analyses for a given subject or calibration should be divided up into three or more

subsets performed on different days and averaged to incorporate day-to-day variability. This also helps insure that the results from a given subject are not lost due to a machine malfunction.

In addition, calibration tips should be extracted and analyzed along with the unknown tips for a given product to minimize any potential effects of storage or long-term analytical drift. This may be more inconvenient for high-throughput analytical laboratories, but ultimately will save time trying to sort out unusual results and re-performing analyses.

Methods based on the analysis of cigarette filters have been used to estimate 'tar' and nicotine yields to smokers. These methods rely on the measurement of filtration efficiencies (FEs) [4]. However FEs may be influenced by both cigarette design features *e.g*., type of filter and levels of filter ventilation, and human smoking behaviour factors such as puff flow-rates and cigarette butt lengths.

The current research conducted by us uses only one method: the whole filter method without the use of smoking machine. The other, a 'part filter' method, analyses a 10 mm section from the mouth end of the filter where the FE remains relatively constant irrespective of puff flow rates and butt lengths but due to time and resources constraints the current research is only focused and limited to use of whole filter method [5].

MATERIALS AND METHODS:

Sampling: The samples for the study were obtained from different areas of Gandhinagar. 10 different brands were taken for the study.

TABLE 1: LIST OF BRANDS OF CIGARETTE SELECTED FOR THE STUDY

S. no.	Name of the brand	Company Name	Cost/stick (INR)
1	Charms®	Vazir Sultan Tobacco Company	5.00
2	Four square®	Godfrey Philips India	5.00
3	Bristol®	Indian Tobacco Company	5.00
4	Gold flake® (brown filter)	Indian Tobacco Company	6.00
5	Navy cut®	Indian Tobacco Company	10.00
6	Wills classic®	Indian Tobacco Company	12.00
7	Marlboro® gold advance	Philip Morris	12.00
8	Gold flake® (white filter)	Indian Tobacco Company	12.00
9	Black®	Djarum (Indonesia)	15.00
10	Dunhill®	British American Tobacco Company	15.00

The samples obtained were collected and stored in air sealed pouches and kept in dark to avoid light exposure. As per the WHO guidelines a minimum of 10 samples is required for such study.

Hence, 10 smoked samples for each brand were used for this study. Total of 100 samples were used for this analysis. The samples selection was done on the basis of the market share of the brand and its cost. The brands selected for the study are listed in **Table 1**.

Physical Study: Following studies were performed for determination of physical parameters of cigarettes samples:

1. Determination of whole cigarette length: It was done by calibrated scale.
2. Determination of filter length: It was done by calibrated scale.
3. Determination of weight of tobacco: It was done by removing out the tobacco from the cigarette and weighing it on electronic weighing machine.

Comparison Microscopic Analysis: The comparative study of smoked and non-smoked filter was done by Leica® comparison microscope. The filters were taken and mounted on the sample holder of the microscope and simultaneously both the samples were studied for determination of physical changes in filter fibres, before and after smoking.

Scanning Electron Microscopic Analysis: The samples of filter fibres were analysed on Zeiss® Scanning Electron Microscope. As the samples were non-conductive, charge effect was given for causing image distortion or drift. The specimen was coated with a thin layer of gold-palladium to provide conductivity and some protection from beam damage. Coating thickness could be several nanometres. After that the samples were mounted with a carbon tape used as a conductive bridge which was connected from the top surface of the sample to sample holder.

UV Spectroscopic Analysis:

Sample Preparation:

Filter Extraction: The smoked filters were extracted with methanol. The filters were separated from the cigarette paper and were transferred to

falcon tube containing 5 mL methanol. The filters were then subjected to vortexing on vortex shaker for 2 min each sample. The samples after vortexing were kept for different time intervals of 5minutes, 30 min, 1 h and 2 h. After the specified time interval, the samples were filtered with Whatman® filter paper and collected in Eppendorf tube.

Preparation of Sample for UV Analysis: From filtered extracts, 100 µL of solution was taken from each extracted solution of methanol by micropipette and transferred to falcon tube. The final volume makeup up to 10 mL was done by methanol in each falcon tube. The final solution so obtained was properly shaken and used for UV spectroscopic analysis.

Preparation of Standard Solution:

Nicotinic acid: It was used as a reference for quantitative determination of nicotine extracted from the smoked cigarette butts. The calibration curve obtained was used to determine the levels of nicotine in filters of smoked cigarettes.

Preparation of Stock Solution: The stock solution was prepared by dissolving 5 mg of nicotinic acid in 50 mL of methanol.

Preparation of Dilutions: The dilutions of various concentrations was prepared by taking 100 µL, 200 µL, 300 µL, 400 µL and 500 µL from the stock solution and making up the volume to 10mL in each dilution.

Quinoline in Methanol: It was used as a reference solution for quantitative determination of tar levels.

Preparation of Stock Solution: The stock solution was prepared by dissolving 5µL of Quinoline in methanol in 50 mL of methanol.

Preparation of Dilutions: The dilutions of various concentrations was prepared by taking 100 µL, 200 µL, 300 µL, 400 µL and 500 µL from the stock solution and making up the volume to 10ml in each dilution.

Procedure for UV spectroscopic Analysis: For the UV spectroscopic analysis Shimadzu-2401 UV Visible Spectrophotometer and Lab India UV 3000+ was used. The cuvettes used were of quartz within the range of 200 – 400 nm. The spectrum, absorption scan mode was used with a slit width of 1.0 cm.

For Quantitative Determination of Nicotine Extracted: All the parameters for analysis were set. The cuvettes were cleaned with methanol. The baseline correction was done by using methanol in both cuvettes. Then the solvent was added in the dry extracts and were shaken and filled in the cuvettes. Followed by which the peak wavelength of absorbance (λ_{max}) of the nicotine alkaloid was determined.

All the methanolic extracted were first analysed followed by the analysis of nicotinic acid reference solution. The calibration curve was plotted for the same.

For quantitative determination of tar levels: Same procedure was adopted for the determination of absorbance at 312 nm for all the methanolic extracts. Followed by which the absorbance of the Quinoline was determined and calibration curve was obtained.

RESULTS AND DISCUSSION:

Results for Physical Study: The results for whole length, filter length and weight of tobacco are as indicated in **Table 2**.

TABLE 2: RESULTS FOR WHOLE LENGTH, FILTER LENGTH AND WEIGHT OF TOBACCO

S. no.	Name of Sample	Whole Length (Avg. Length of 10 samples) (mm)	Filter Length (Avg. Length of 10 samples) (mm)	Weight of tobacco (Avg. Weight of 10 samples) (gm)	Cost/ stick (INR)
1	Charms®	70	17	0.630	5.00
2	Four square®	68	16	0.570	5.00
3	Bristol®	70	15	0.601	5.00
4	Gold Flake® (brown filter)	70	15	0.635	6.00
5	Navy Cut®	75	26	0.663	10.00
6	Wills Classic®	85	25	0.615	12.00
7	Marlboro® gold advance	85	27	0.625	12.00
8	Gold Flake® (white filter)	85	27	0.649	12.00
9	Black®	85	25	0.630	15.00
10	Dunhill®	95	28	0.571	15.00

Whole Length: The cigarettes with the lower cost Charms®, Four Square®, Bristol® and Gold flake® (Brown Filter**)** had similar length of 70 mm as shown in **Table 2**. It is clearly observed that with the increase in the cost, there is consistent increase in the whole length of cigarettes different brands. The maximum length observed was 95mm for Dunhill® cigarette brand having cost of price of INR 15.00/stick. The minimum whole length of 68 mm was observed for Four square® brand having a cost price of INR 5.00/stick.

Filter Length: The cigarettes with lowest cost had smaller filter length averaging 16mm. However, with the increase in the cost by INR 4.00, from Gold Flake® (Brown) to Navy Cut® there was sudden increase in length by 10 mm. The maximum filter length was found to be of Dunhill® with a cost of INR 15.00. Therefore, on an average, there was a direct relationship between the whole length and filter length of cigarette with respect to cost price as seen in **Table 2**.

Weight of Tobacco: The average weight of tobacco in all brands was found to be 0.618gms. The weight of tobacco in all cigarettes was found to be similar ranging from 0.601 gm to 0.663 gm. Two brands, Dunhill® and Four Square® were found to similar and lowest content of tobacco weighing 0.571 and 0.570 gm respectively. Therefore, as given in **Table 2**, there was hardly any difference between the weight of tobacco and cost price of different brands of cigarette.

Results for Comparison Microscopy: The comparison microscopic study of the filters showed a complete change in the physical state of the filters of each brand. The filters before smoking were found to be clear and white in its appearance, however after smoking there was a complete change in the physical appearance of the filter as seen in **Fig. 1 - 10**. The filter fibre after smoking were completely stained with the smoke content.

Presence of Adsorbent: Only two cigarette brands, Dunhill® and Marlboro® were found to have activated charcoal particles towards the tip of the mouth end of the filter as seen in figure Presence of adsorbents in the filter increases the entrapment of the smoke particles. It was also observed that there was an increase in the staining of filter fibre n filter with adsorbent as compared to other brands without adsorbent as seen in **Fig. 1 - 10**. Also the presence of adsorbents was found only in cigarettes with higher cost as indicated in **Table 3**.

TABLE 3: RESULT FOR DETERMINATION OF PRESENCE OF ADSORBENT

S. no.	Name of sample	Presence of adsorbent before smoking (Activated charcoal)	Cost/stick (INR)
1	Charms®	Nil	5.00
2	Four square®	Nil	5.00
3	Bristol®	Nil	5.00
4	Gold flake® (brown filter)	Nil	6.00
5	Navy Cut®	Nil	10.00
6	Wills Classic®	Nil	12.00
7	Marlboro® gold advance	Present	12.00
8	Gold Flake® (white filter)	Nil	12.00
9	Black®	Nil	15.00
10	Dunhill®	Present	15.00

Results for Scanning Electron Microscopy:

Presence of Adsorbed Particles After Smoking: In the scanning electron microscopic images obtained, it is clearly observed that the filter fibre of non-smoked cigarette had no particles adsorbed on its surface as shown in **Fig. 11**. The surface of filter fibre was clean with complete absence of smoke particles.

The filter fibre of cigarette with the lowest cost (Charms®) was found to have considerably less number of smoke particles adsorbed on the filter surface as shown in **Fig. 12**.

The filter fibre of cigarette with intermediate cost (Navy Cut®) had more number of particles on its filter surface but comparatively less than that of cigarette with highest cost as shown in **Fig. 13**.

The Filter fibre of cigarette with the highest cost (Dunhill®) clearly revealed the presence of highest number of smoked particles adsorbed on the fibre surface as shown in **Fig. 14**. On the basis of above results obtained, it is clearly observed that as the cost of cigarette increases, the entrapment efficiency of the filter also increases thus indicating that costlier the cigarette, higher is its filter's efficiency to retain the toxic compound of tobacco smoke as seen in **Fig. 11-14**.

Result for UV Spectroscopic Study: The analysis of nicotine in each cigarette brand was done by UV spectroscopic analysis for both non-smoked tobacco and smoked filter. For the tobacco obtained from the non-smoked cigarette, it was found that each brand has shown the presence of nicotine at 260 nm. The maximum absorbance was observed for the Wills Classic® brand and minimum absorbance was observed for Charms® brand as seen in **Table 4**.

TABLE 4: UV ABSORBANCE OF TOBACCO OBTAINED FROM EACH NON-SMOKED CIGARETTE STICK AT 260 nm

S. no.	Name of the sample	Absorbance at 260nm (Abs)	Cost/stick (INR)
1	Charms®	0.6150	5.00
2	Four Square®	0.9798	5.00
3	Bristol®	0.8683	5.00
4	Gold Flake® (brown filter)	0.7881	6.00
5	Navy Cut®	0.8120	10.00
6	Wills Classic®	0.9877	12.00
7	Marlboro® gold advance	0.9375	12.00
8	Gold flake® (white filter)	0.7550	12.00
9	Black®	1.0352	15.00
10	Dunhill®	0.8401	15.00

The average UV absorbance of nicotine from smoked filters of 10 samples of each brand at 260 nm was observed as shown in **Table 5**.

TABLE 5: AVERAGE UV ABSORBANCE OF SMOKED CIGARETTE FILTERS OF 10 SAMPLES OF EACH BRAND

S. no.	Name of the brand	Average absorbance from 10 samples (Abs)
1	Charms®	0.3453
2	Four square®	0.1907
3	Bristol®	0.5023
4	Gold flake® (brown filter)	0.2964
5	Navy Cut®	0.5524
6	Wills Classic®	0.3533
7	Marlboro® gold advance	0.4100
8	Gold flake® (white filter)	0.6804
9	Black®	0.6619
10	Dunhill®	0.8920

The absorbance of Nicotinic Acid standard solution at 262 nm was obtained as seen in **Table 6**.

TABLE 6: ABSORBANCE OF NICOTINIC ACID STANDARD SOLUTIONS

S. no.	Sample Quantity (ppm)	Absorbance (Abs)
1	100	0.2043
2	200	0.3749
3	300	0.5221
4	400	0.6633
5	500	0.8587

The UV spectrum for nicotinic acid standard at 261 nm was obtained as seen in **Fig. 1**.

Calibration curve for nicotinic acid standard solutions was plotted as seen in **Table 7**. The R^2 was found to be 0.997. Absorbance of smoked samples of cigarettes with reference to standard nicotinic acid calibration curve was plotted as seen in **Fig. 1** and **Table 8.**

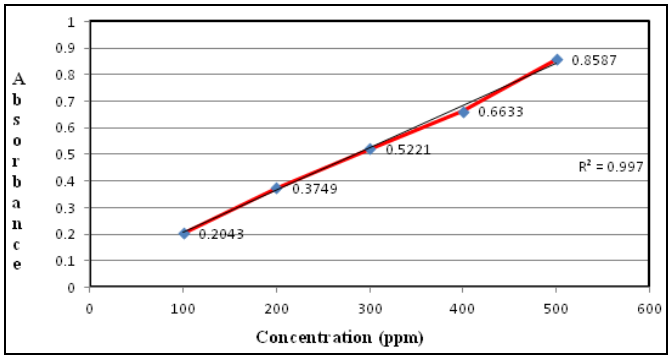

FIG. 1: CALIBRATION CURVE FOR NICOTINIC ACID STANDARD SOLUTION

The Dunhill® Brand was known to have maximum absorbance (0.8920), hence was observed to retain maximum concentration of nicotine in its filter as seen in **Fig. 2**. The Four Square® brand was known to have minimum absorbance (0.1907), hence was observed to retain minimum concentration of nicotine in its filter as seen in **Fig. 2**.

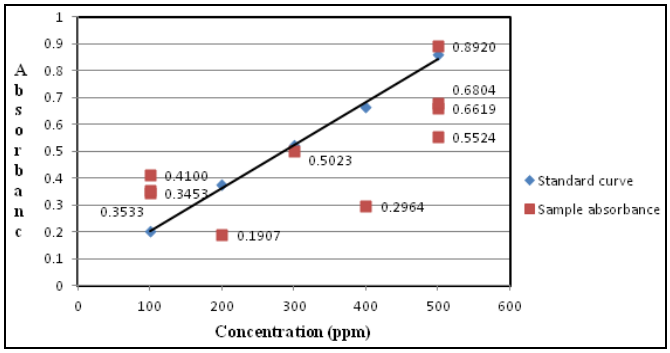

FIG. 2: ABSORBANCE OF SMOKED SAMPLES OF CIGARETTES WITH REFERENCE TO STANDARD NICOTINIC ACID CALIBRATION CURVE

The concentration of the nicotine in the smoked samples of different brands of cigarettes with reference to nicotinic acid was found to have linear relationsship with respect to the cost price of cigarette as shown in **Fig. 1**. The UV spectrum for quinoline in methanol was obtained as seen in **Fig. 3**.

The absorbance of Quinoline in methanol at 313nm was obtained as seen in **Table 7**.

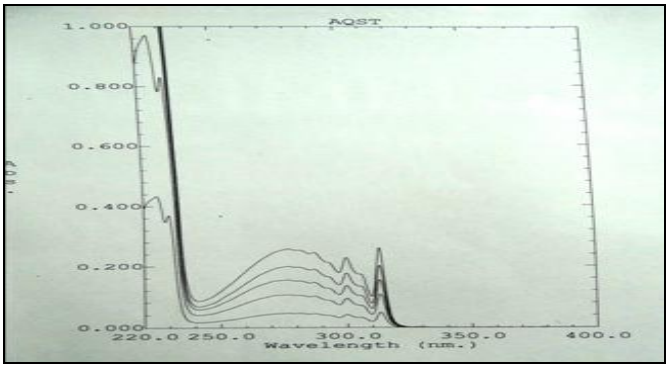

FIG. 3: UV SPECTRUM OF STANDARD SOLUTIONS OF QUINOLINE IN METHANOL

TABLE 7: ABSORBANCE OF QUINOLINE IN METHANOL

S. no.	Sample Quantity (ppm)	Absorbance (Abs)
1	100	0.0489
2	200	0.1077
3	300	0.1553
4	400	0.2030
5	500	0.2610

The Claibration curve was plotted for Nicotinic Acid standard solution as seen in **Fig. 4**. The R^2 value was found to be 0.9983.

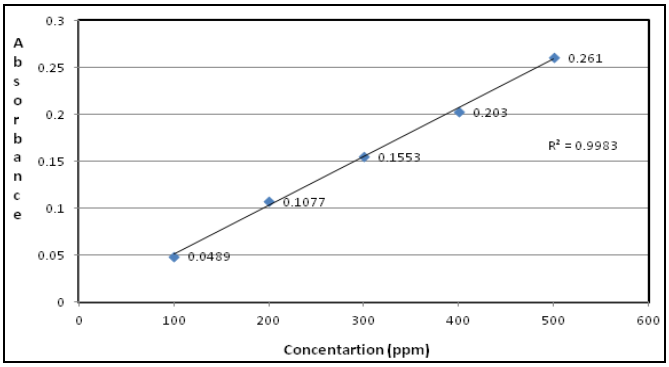

FIG. 4: CALIBRATION CURVE FOR NICOTINIC ACID STANDARD SOLUTION

TABLE 8: ABSORBANCE OF SMOKED SAMPLES OF CIGARETTES AT 313nm

S. no.	Name of the brand	Absorbance
1	Charms®	0.0970
2	Four square®	0.1230
3	Bristol®	0.1260
4	Gold flake® (brown filter)	0.1290
5	Navy cut®	0.1420
6	Wills classic®	0.2640
7	Marlboro® gold advance	0.3580
8	Gold flake® (white filter)	0.4400
9	Black®	0.4680
10	Dunhill®	0.8510

Absorbance of smoked samples of cigarette with reference to that of Quinoline in methanol is obtained as seen in **Fig. 5**. The Dunhill® Brand was known to have maximum absorbance (0.8510) with reference to quinoline in methanol, hence was observed to retain maximum concentration of TAR in its filter as seen in **Fig. 5**.

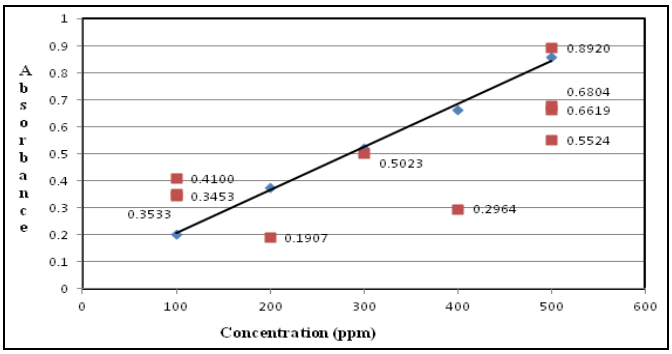

FIG. 5: ABSORBANCE OF SMOKED SAMPLES OF CIGARETTES WITH REFERENCE TO STANDARD QUINOLINE IN METHANOL CALIBRATION CURVE

The Charms® brand was known to have minimum absorbance (0.0970), hence was observed to retain minimum concentration of nicotine in its filter as seen in **Fig. 5**. The concentration of the TAR in the smoked samples of different brands of cigarettes was found to have linear relationsship with respect to the cost price of cigarette as shown in **Fig. 5**.

CONCLUSION: The present study was focused on the determination of filtration efficiency of the filter to retain toxic compound: nicotine and tar. Apart from this, the filter lengths were digitally compared and a comparative study of the compound retained by the filter was done by the comparison microscope and SEM. Since the subject is very vast, the current research was focussed on the estimation of the nicotine and tar by UV spectroscopic analysis from the smoked filters. On the basis of the physical study performed, it can be concluded that the whole length and the filter length of the cigarette has a direct relationship with the cost price of the cigarette. With the increase in the whole and filter length, there is an increase in the cost of the cigarette.

On the basis of the comparison and Scanning Electron Microscopic study, which are sophisticated techniques for physical study having a greater resolution, it was clearly observed that the cigarette with the higher cost was able to retain more toxic compounds as compared to that of cigarette with lower cost. Hence, this part of research also showed a direct relationship between the cost and retaining efficiency of the filter.

On the basis of the UV spectroscopic analysis, an advanced technique for the quantitative study of nicotine and tar with reference to that of nicotinic acid and quinoline revealed a direct relationship of the filtration efficiency of the filter with respect to that of the cost. The cigarettes with higher costs were able to retain more toxic compounds as compared to that of cigarettes with lower cost. Hence, it can be concluded that increase in the cost of the cigarette, increases the filtration efficiency of the filter of the cigarette.

The other instrumental techniques which are equally competent for the study of the other toxic compounds GC, HPLC, UPLC and elemental analysis could be better achieved by AAS, ICP and EDXRF.

With the current research, we can conclude that the cost of the cigarette brand is an essential parameter for determining the quality of the cigarette brand. It was found that higher the cost of cigarette, the greater is the filtration efficiency of the filter to retain the toxic compounds. However, further study is still required in this field to obtain an in depth analysis of filtration efficiency of the filters. The future study is required in the field of validation of the methods for the identification of various other the toxic compounds such as PCB, Benzaldehyde, ammonia, benzene, benzopyrene, acryldehyde, formaldehyde and carbon mono oxide.

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CONFLICT OF INTEREST: The authors do not have any conflict of interest amongst them.

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