Studies on physicochemical and deteriorated properties of different brands of mustard oils at room temp and after heated at 180^0C

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***Abstract*: Oils and fats are valuable necessary nutrients that play an important role in human life and health. Rancidity oil is generally used to denote unpleasant odours and flavours in foods resulting from deterioration in the fat or oil portion of a food. The studies aimed at assessing during storage at room temperature and the effect of heat treatment at 180^0C for 30 minutes and evaluate the oxidative status and physicochemical properties of edible oils of different brands of Mustard oils sold in local market. The quality of different brands of commonly used in cooking Mustard oils was analyzed by evaluating physicochemical properties. The experimental results obtained that, RI and specific gravity and colour were in the range of 1.4650-1.4681, 0.9012-0.8940 and1.34-10.82 before and after frying. AV and PV were ranged between 0.44-3.25 in fresh oils,0.85-6.84in after frying and 0.61-3.91and 1.34-6.80 mg KOH/g before and after frying oil while SV and EV were ranged between 171.58-173.82 and 170.82-172.8 mg KOH/g oil in fresh oils and in between 179.36-178.57 and 173.4-178.39mg KOH/g oil in after frying oils respectively. P-AV and TV were in the range of 1.42-6.82 and 2.64-11.43 in before frying while in case of after frying it is 2.4-8.82 and 5.32-18.74 meq/kg respectively. In general, after frying, RI, colour, AV, PV, P-anisidine and totox values were found higher and specific gravity and iodine values are lower than as compared with safety limit. In case of AV in after frying are increases and is above 5 mg KOH/gm of oil as compared to codex limit and it deviates food safety standard. The results showed degradation in the physico-chemical properties of the vegetable cooking oil after frying.**

***Keywords*: Mustard oil, heat treatment, oxidative and physicochemical properties**

1. Introduction

Vegetable oils are important in human nutrition as they provide energy, essential fatty acids and facilitate the absorption of fat-soluble vitamins [1]. Jambunathan et al(2008) studied and stated that oil quality and its stability are therefore very important for the consumer's desirable use usually as cooking ingredient [2]. the quality of any oil is indicated by some physicochemical properties which provide an indication of both the nutritive and physical quality of the oil. These properties include iodine value, peroxide value, saponification value, free fatty acid, colour appearance etc. for retaining oil quality, care must be taken when storing vegetable oils for a period of time to prevent their deformation as they easily undergo oxidative deterioration, hence shortening their shelf-life.

Fats and oils are parts of normal daily consumptions. As a major source of energy, fats and oils are considered as important nutrients in human diets. The edible oils are used in cooking as well as in traditional medicine for the treatment of colds, coughs, bronchitis, edema and burns, also play an important role in the body as carriers of essential fatty acids which are not synthesized in the body but are needed through the diet to maintain the integrity of cell membranes. They are also needed for the synthesis of prostaglandins which have many vital functions to perform in the body [3-4].Vegetable oils are beneficial and popular due to their cholesterol-lowering effect. In contrast to animal fats, which are predominantly saturated and hence do not react readily with other chemicals, especially oxygen, unsaturated vegetable oils are more reactive. Vegetable oils are essential in global nutrition depending on the regional conditions, a variety of oils are produced in different qualities [3-5].

Mustard oil from *Brassica nigra* has 30% protein, calcium, phytins, phenolics and natural anti-oxidants. Mustard oil contains a high amount of mono-unsaturated fatty acids and a good ratio of polyunsaturated fatty acids, which is good for the heart. Mustard oil contains the least amount of saturated fatty acids, making it safe for heart patients. Rancidity is a term generally used to denote unpleasant odours and flavours in foods resulting from deterioration in the fat or oil portion of a food. Three different mechanisms of rancidity may occur. Lipids and triacylglycerol naturally occur in oils and fats. Their chemical composition contains saturated and unsaturated fatty acids and glycerides.

1.1 Literature review

Thermal, oxidative and hydrolytic decomposition of the oil may occur, when oil or fat is exposed to air, water and heat.Fats and oils are oxidized to form hydroperoxides, the primary oxidation products. These peroxides are extremely unstable and decompose via fission, dehydration, and formation of free radicals to form a variety of chemical products, such as alcohols, aldehydes, ketones, acids, dimers, trimers, polymers, and cyclic compounds [6,7].

Zagir and co-workers,studied the physicochemical properties like density, viscosity, boiling point, saponification value (SV), iodine value (IV),and peroxide value (PV) of Corn and Mustard oils were studied to evaluate the compositional quality of oils and also to investigate the effect on the use of same oil for repeated frying as it ultimately changes the physicochemical, nutritional and sensory properties of the oil. FT-IR spectroscopy was used to evaluate the degree of oxidation after heating and frying processes. Results revealed that due to the temperature change in the oil there is a notable difference in the spectral band which showed that the proportions of the fatty acids were changed. The spectra of Corn oil at the boiling point and at multiple frying times with a piece of potato showed frequencies in range of 2852.7–2926.0 cm_1 while in Mustard oil an additional peak was observed at 3633.8 cm_1 which exhibits the secondary oxidized product formation (Zahir et al(2014)[8].

The increasing use of edible oils and fats for the preparation of fried products requires an increasingly strict control in order to maintain the quality and safety of the fried foods. Due to the knowledge that the frying process alters the chemical nature of the heated oil and its consumption represents health risks [9,10].

Repeated frying causes overall oxidative and thermal reactions which results in change in the physicochemical, nutritional and sensory properties of the oil. Deep frying is one of the most common methods used for the preparation of food. [11]. In frying, due to hydrolysis, oxidation and polymerization processes the composition of oil changes which in turn changes the flavour and stability of its compounds [12]. In deep frying different reactions depend on some factors such as replenishment of fresh oil, frying condition, original quality of frying oil and decrease in their oxidative stability [13]. Atmospheric oxygen reacts instantly with lipid and other organic compounds of the oil to cause structural degradation in the oil which leads to loss of quality off odour and is harmful to human health [14]. Therefore, for retaining the quality of oil, it is essential to monitor the quality of oil to avoid the use of abused oil due to the health consequences of consuming foods fried in degraded oil, to maintain the quality of fried foods and to minimize the production costs associated with early disposal of the frying medium [15].
Santos et al(2005)stated that deep frying is the most common and one of the oldest methods of food preparation worldwide. It involves heat and mass transfer. To reduce the expenses, the oils tend to be used repeatedly for frying. When heated repeatedly, changes in physical appearance of the oil will occur such as increased viscosity and darkening in colour , which may alter the fatty acid composition of the oil. Heating causes the oil to undergo a series of chemical reactions like oxidation, hydrolysis and polymerization. During this process, many oxidative products such as hydroperoxide and aldehydes are produced, which can be absorbed into the fried food [16].Several researcher studied that the chronic consumption of repeatedly heated vegetable oils could be detrimental to health and found an independent positive association between the risk of hypertension and intake of heated cooking oil. These accumulating data suggest chronic intake of heated cooking oils increases the risk of cancer and cardiovascular diseases [17,18].

1.2 Physicochemical properties and their importance

Physicochemical properties of oils are determined to know the quality, purity and identification. Characteristic properties are properties that depend on the nature of the oil. These are used to characterize oil, irrespective of location or sources of origin [19]. Specific gravity is determined and calculated at temperature 20°C as a ratio of mass in air of a given volume of the oil or fat to that of the same volume of at 20°C [20].Refractive index is the degree of refraction of a beam of light that occurs when it passes from one transparent medium to another. The refractive index of an oil can be estimated with the aid of a refractometer in degrees, at 20°C usually. The value obtained is unique for a particular oil and can therefore be used to check adulteration and purity of oil [21]. Free fatty acid value is often used as general indication of the condition and edibility of oils[22]. Iodine value is a measure of the degree of unsaturation or double bonds among the fatty acid present in the oil therefore it does not tell precisely the fatty acids composition of any oil. Iodine value or number is useful as a guide to check adulteration of oil and also as a process control of oil[22]. Saponification value is a rough index of the molecular weight of the fat or oil. The smaller the saponification value the higher the molecular weight. It also indicates the quantity of alkali required for conservation of a definite amount of fat or oil into soap. It is used to check the adulteration of fat and oils[20]. Esterification value is the difference between the saponification value and the free fatty acid value of the fats and oils. Peroxide value is a measure of peroxides contained in the oil and used in determining the degree of spoilage. The standard peroxide value for edible oils which have not undergone rancidity must be well below10 meq/kg [22].Other variable properties of oil include p-anisidine value, solubility, freezing point, colour, odour and boiling point. Oil spoilage can be prevented through exclusion of air, addition of antioxidants, addition of chelation agents and hydrogenation[23].

The Objective of research study are to assessing the different brands of mustards oils during storage at room temperature and the effect of heat treatment at 180^{0}C at different hours and evaluate the oxidative status and physicochemical properties of edible oils of different brands of Mustard oils sold in local market.

2. Material and Methods

2.1 procurement of samples

For study the effect of heating, The different brands of communally used mustard oil Refined Mustard oil, Pure Kacchi Ghaani Mustard oil, Kacchi Ghaani Mustard oil, Mustard oil, Mustard oil, Kacchighani mustard oil, Kacchighani mustard oil, refined mustard oil were used, the samples were taken randomly and each one was heated in three aluminum containers, at 180°C for 30 minutes and the analysis were carried out for study the effect of heatihg on Mustard oil, Refractive indices of all samples were determined at 20-25°C by Abb-Refractometer according to AOAC (1984),colour measured on Lovibond Tintometer, for viscosity measurement using Hakke viscometer [24]. Acid value, Iodine value, Saponification value ,Ester value,peroxide numberand P-anisidine values were determined according to AOAC (1984) [24].

2.2 Experimental procedures [24-27,29-37]
2.2.1 Determination of physical properties
2.2.1.1 Determination of Refractive index
The refractive index (RI) of the oil sample was measured using Abbe refractometer following IS 548 (IS 548, 2015a) method.
2.2.1.2 Determination of colour
Lovibond tintometer was used to measure the colour of samples as described in AOCS method 13e-92 (AOCS, 1989a).Colour was reported in terms of Lovibond units as:- Colour reading = (a Y + 5 b R) Where, a = sum total of the various yellow slides (Y) used b = sum total of the various red (R) slides used Y + 5R is the mode of expressing the colour of oils

2.2.1.3 Determination of Specific Gravity of the Oil

The specific gravity of the oil samples was determined using a 25 ml specific gravity bottle. The specific gravity bottle was weighed empty and then filled with the oil sample up to the mark on the bottle. The bottle with oil sample was weighed again. The weight of the empty bottle was subtracted from the total weight of the bottle and oil sample. The weight of the empty bottle was divided by the total weight of the bottle and oil to obtain the weight of the oil sample. The weight of the oil sample was then divided by the weight of an equal volume of water to get the specific gravity of the oil sample.

2.2.1.4 Determination of viscosities
Measured Viscosity of various brands of vegetable oil before and after frying using Haake Rheowin Viscometer: flow characteristics for viscosity, Take 50ml sample of the original oil in the container set the instrument with water circulating for control of temperature thermostat as per manuals, the viscosity determine at the 27 ^{0}C temperature. Take the reading of pure vegetable oils before and after frying which are shown in Table 1-2.
2.2.2 Determination of Chemical properties
2.2.2.1 Determination of Acid value : Acid value was measured by titration with sodium hydroxide according to the American Oil Chemists' Society (AOCS) method Cd 8-53 (AOCS, 1989b).
2.2.2.2 Determination of Iodine value: The iodine value (IV) of the sample was measured using IS 548 (IS 548, 2015c).
2.2.2.3 Determination of Peroxide value: The acetic acid-chloroform method of the AOCS (Method Ca 5a-40) was used for determination of the peroxide value (PV) (AOCS, 1989c).
2.2.2.4 Saponification value: The saponification value (SV) of the sample was obtained by following IS 548 method (IS 548, 2015b).
2.2.2.5 Method for determination of ester value

The ester value is the 'mg' of KOH required to react with glycerol/ glycerin after saponify 1 g of oil sample. Ester value is calculated by the following relation

Ester Value = Saponification Value – Acid Value

2.2.2.6 Determination of p-anisidine Value: The p-anisidine value was obtained using a double beam spectrophotometer (UV 2300, LabIndia) according to the AOCS method Cd 18-90 (AOCS, 1989d).
2.2.2.7 Determination of Totox value
The totox (i.e. total oxidation products) value was calculated by AOCS Ccl3e-92 method. The Totox value was calculated as: **Totox value=TV = (2 x PV) +AV**

4.0 Experimental results of physicochemical characteristics of different brands of soyabean oil before and after frying.

Table1. Measured Physicochemical characteristics of Mustard oil storage at room temperature before frying.

Sr. no	Code	RI*	Sp. Gravity	Colour (red units)	Viscosity Cp	AV mg KOH / g oil	IV gI2/100 g	PV Meq/ Kg	SV mg KOH / g oil	EV mg KOH /g oil	P-anisidine	Totox Value
1	Rmu	1.4650	0.9012	2.48	34.19	0.54	99.33	0.61	172.68	172.14	1.42	2.64
2	PKgmu	1.4652	0.9219	6.80	41.82	3.02	101.52	3.91	175.82	172.8	4.70	12.52
3	Kgmu	1.4653	0.9312	6.38	43.38	3.25	102.48	2.4	174.52	171.27	4.84	9.64
4	Mu	1.4655	0.9123	5.28	39.20	2.13	98.28	3.24	173.68	171.55	5.92	12.4
5	Rmu1	1.4652	0.9120	1.34	34.86	0.44	102.28	0.98	171.58	171.14	1.90	3.86
6	PKgmu1	1.4654	0.9214	5.82	38.36	3.25	103.40	3.74	174.38	171.13	6.82	14.3
7	Kgmu1	1.4658	0.9218	4.28	38.24	3.23	102.36	2.8	173.82	170.59	5.83	11.43
8	Mu1	1.4660	0.9148	5.92	40.26	2.67	104.92	2.9	172.94	170.27	5.62	11.42
9	Total	11.7234	7.3366	38.3	310.31	18.53	814.57	20.58	1389.42	1370.89	37.05	78.21
10	Mean	1.4654	0.9171	4.79	38.79	2.32	101.82	2.57	173.68	171.36	4.63	9.78
11	SD	0.0005	0.001	0.05	0.47	0.26	0.28	0.74	0.37	0.83	0.3	0.28
12	CV	0.04	0.13	1.1	1.22	11.4	0.28	28.98	0.21	0.49	6.55	2.86
13	SEM	0.0003	6.94	0.03	0.27	0.15	0.16	0.43	0.21	0.48	0.17	0.16

(*RI-Refractive index, AV-acid value, IV-Iodine value, PV-peroxide value, SV-Saponification value, EV-Ester value, SD-standard deviation, CV-coefficient of variation, SEM standard error)

Table 2. Measured Physicochemical characteristics of mustard oil after frying.

Sr. no	Code	RI*	Sp. Gravity	Colour (yellow +red units)	Viscosity Cp	AV, mg KOH / g oil	IV gI2/100 g	PV, Meq/ Kg	SV, mg KOH / g oil	EV, mg KOH /g oil	P-anisidine	Totox Value
1	Rmu	1.4679	0.8859	3.80	81.28	0.97	92.38	1.34	179.36	178.39	3.20	5.88
2	PKgmu	1.4673	0.8924	9.84	88.42	6.63	91.88	6.24	182.46	175.83	7.22	19.7
3	Kgmu	1.4674	0.8798	10.82	89.36	6.73	94.20	5.64	182.12	175.39	7.46	18.74
4	Mu	1.4676	0.8830	9.82	90.18	5.38	91.68	6.80	181.84	176.46	8.82	22.42
5	Rmu1	1.4674	0.8828	4.20	78.98	0.85	96.20	1.46	179.42	178.57	2.4	5.32
6	PKgmu1	1.4675	0.8940	10.40	92.38	6.42	100.82	6.15	181.91	175.49	9.9	22.2
7	Kgmu1	1.4679	0.8896	9.40	91.48	6.84	96.38	4.80	180.24	173.4	8.8	18.4
8	Mu1	1.4681	0.8810	9.80	92.12	5.39	98.24	6.01	179.94	174.55	9.0	21.02
9	Total	11.74	7.0885	68.08	704.2	39.21	761.78	38.44	1447.29	1408.08	56.8	133.68
10	Mean	1.4676	0.8861	8.51	88.03	4.90	95.22	4.81	180.91	176.01	7.1	16.71
11	SD	0.0007	0.0008	0.32	0.28	0.35	0.58	0.3	0.41	0.25	0.51	0.45
12	CV	0.05	0.09	3.73	0.32	7.1	0.61	6.29	0.23	0.14	7.51	2.7
13	SEM	0.0004	4.62	0.18	0.16	0.2	0.34	0.17	0.24	0.14	0.29	0.26

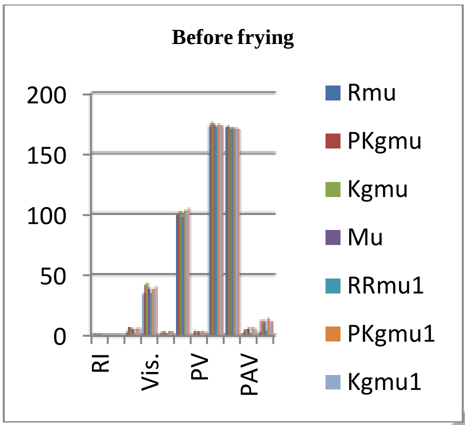
(*RI-Refractive index, AV-acid value, IV-Iodine value, PV-peroxide value, SV-Saponification value, EV-Ester value, SD-standard deviation, CV-coefficient of variation, SEM standard error)

Table 3.Test results of frying oils and codex standards

Sr.no	Test	Before frying	After frying	Codex Alimentarius commission standard	FSSAI
1	RI	1.4650-1.4660	1.4673-1.4681	--	---
2	Specific gravity	0.9012-0.9312	0.8798-0.8940	0.899-0.920	0.91-0.93
3	Colour(yellow+red units)	1.34-6.8	3.80-10.82	--	--
4	Viscosity(Cp)	34.19-43.38	78.98-92.38	--	28.8c St-35.2cSt
5	Acid value	0.44-3.25	0.85-6.84	0.6-5.0 mg KOH/g	0.5-6.0 mg KOH/g
6	Iodine value gI2/100g	98.28-104.92	91.88-100.82	--	--
7	Peroxide value	0.61-3.91	1.34-6.80	10.0 $MeqO_{2/Kg}$	1-10 $MeqO_{2/Kg}$
8	Saponification value	171.58-173.82	179.36-178.57	--	--
9	Ester value mg KOH/g	170.82-172.8	173.4-178.39	--	---
10	P-anisidine value	1.42-6.82	2.4-8.82	20 $MeqO_{2/Kg}$	--
11	Totox value	2.64-11.43	5.32-18.74	26 $MeqO_{2/Kg}$	--

5. Statistical Analysis

The data obtained from the experimental measurements and accuracy of different parameters for different brands of mustard oils have been analysed and the Statistical parameter like standard deviation, coefficient of variance and standard mean error were calculated for RI, colour, specific gravity, viscosity, AV, IV, PV,SV, EV, P-anisidine and totox values for different brands of mustard oils before and after frying. All the experiment was carried out in triplicate and the results are presented as the mean ± SD, CV, ± SEM. Accuracy and descriptive Statistics of different oils from different parts of India as shown in figure1to3.



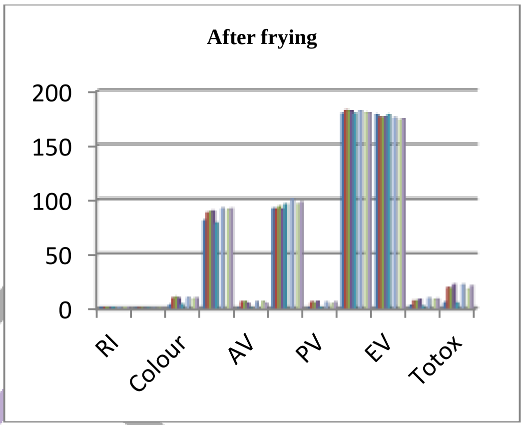


Fig 1-2 shows that plot of obtained results of different parameter of different Mustard edible vegetable oils before and frying.

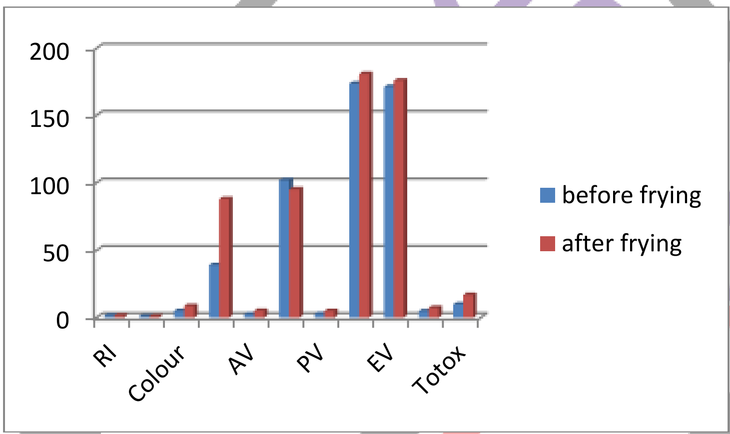


Fig.3 Accuracy and descriptive Statistics of different Mustard oils from different parts of India as shown in figure3.

6. Results and discussion

The studied properties of different brands of Mustard oil samples determined the quality of these samples. Lipid oxidation is one of the major causes of food spoilage. It is of great economic concern to the food industry because it leads to the development, in edible oils and fat-containing foods, of various off-flavors and off-odors generally called rancid, which renders these foods unacceptable or reduces their shelf life [28]. All results determined are shown in Table 1-2.When the different brands of Mustard oil samples obtained from the local markets have been examined before and after frying for physiochemical parameters such as RI, colour, specific gravity, viscosity, AV, IV, PV,SV, EV, P-anisidine and totox values were used to monitor deterioration of fried oil while it was used to authenticate oil samples. The obtained results of the different brands of Mustard frying oil samples are compared to international standards before frying and after frying are illustrated in Tables 1-2 and figures 1-3. In this study, It is seen that, the results showed that in the range of 1.4650-1.4681(±0.00-0.00) in case of RI which are exceeded, In case of specific gravity, it is in the range of 0.9012-0.8940(±0.001-0.00) which are decreased as compared with codex safety standards. Colour in the yellow units in the range of 1.34-10.82(±0.005-0.32) while in case of acid value, it is seen in range 0.44-6.84(±0.26-0.35) which are increased. In case of IV,98.28-100.82(±0.28-0.58)which are gradually decreased as the result of destruction of double bonds in after frying, the result was not in this expectation, while in case of PV it is in the range of 0.61-6.8(±0.74-0.3)which are lower as compared with codex limit of 10 meq/kg in after frying, The P-anisidine value are in between 1.42-8.82(±0.3-0.51) which are within the limit of 20 meq/kg in case of after frying, The totox value are in the range of 2.64-18.74(±0.28-0.45) which are also within the limit of 26 meq/kg and conforms the codex standards. SV and Ester value are in the range of 171.58-178.57(±0.37-0.41) and 170.82-178.39(±0.83-0.25) respectively. The AV, PV,P-Anisidine and totox values which were significantly lower as compared to the Codex Alimentarius Commission standards in after frying. In general, It can be noticed that, after frying, viscosities, AV,PV,P-anisidine and totox values were found within safety limit and specific gravity and iodine values are lower than as compared with safety limit. The results showed that all the physico-chemical properties of all the mustard vegetable cooking oil samples before and after frying were close to international standards except acid value standard. A.V. measures the content of free fatty acids formed upon the hydrolytic degradation of lipid molecules, thus contributing the reduction of shelf life of oil. According to Codex

Alimentarius Commission standard acid value up to 5 mg KOH/gm of oil is safe for consumption. The acid value (table 1)in before and after frying is above 5 mg KOH/gm of oil so it is deviates food safety standard.

6. Conclusion

The experimental results of different brands of Mustard oil before and after frying are investigated. It is clear from the obtained results that, before and after frying, RI, colour, viscosities, AV,PV,P-anisidine and totox values were found within the safety limit except AV and specific gravity and iodine values are lower than as compared with safety limit. The results showed that first stage of oxidation degradation in the physico-chemical properties of the vegetable cooking oil after frying due to AV are increased in after frying. AV,PV, P-anisidine and totox values of edible oil increased after frying compared with that before frying (might be due to the effect of high frying temperature causing destruction of some glycerides). The study concluded that most of the vegetable oils are susceptible to oxidation at higher temperature. It is important to add antioxidants such as vitamin E as preservatives in vegetable oils to slow down the rate of oxidation.

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