

## ORIGINAL RESEARCH ARTICLE

# Study of vinyl group effect on thermal and mechanical properties of some polymers and silicone rubber

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### ABSTRACT

The research presented in this article has focused on the identification of the potential use of two methods: weight change analysis and swelling-weight balancing, to study polymer and silicone rubber samples at consistent temperatures for an extended period. This ensures the stability of these materials for future industrial applications. Throughout the research, it was determined that these methods allow for simultaneous observation of various processes to which the samples were exposed, including degradation and thermal oxidation. The analysis of the obtained results has indicated that a sample made of methylvinylsilicon rubber containing 2% vinyl by weight exhibited superior properties compared to other samples. These properties include a decrease in the rate of rubber chain bond breakage, resistance to various solvents, and improved mechanical characteristics.

**Keywords:** methylvinylsiloxane; dimethylsilicon, methylsilicon; degradation; polymer chain; vulcanization

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## 1. Introduction

Silicones are known as inorganic polymers that have a chemical structure similar to silicic acid and inorganic silicates. Their chains contain Si-O-Si bonds, which give them high chemical and thermal resistance. In addition, silicones can have inorganic hydrogen or organic methyl, vinyl, and other groups attached to the silicon atoms. The demand for polymers and silicone rubber is high globally, as they are used in various industrial fields. These materials have a specific set of chemical, mechanical, and electrical properties, similar to other polymer compounds. The key properties of organosilicon polymers include thermal stability in a temperature range from  $-55^{\circ}\text{C}$  to  $+250^{\circ}\text{C}$ , resistance to oxygen and ozone, hydrophobicity, electrical insulation, low viscosity coefficient with temperature, and low surface tension<sup>[1-3]</sup>.

The crosslinking of silicone rubber is made possible by the reactivity of the vinyl group present in the MVQ and PMVQ polymer rubber chains. These vinyl groups are activated by crosslinking agents. The process itself is highly dependent on the specific type of chemical agent used. Crosslinking can occur at varying rates, resulting in complete or incomplete crosslinking, and can lead to irregular polymer structures. The filler used in silicone rubber is completely inert and

serves to stabilize the polymer framework formed during crosslinking

Thermoanalytical methods are suitable for monitoring the degradation process in both air and vacuum environments, as well as studying thermooxidation reactions that occur simultaneously with degradation in the presence of air.

a) Varying the mode of thermoanalytical measurements and experimental parameters can provide valuable information to industrial specialists interested in thermal stability, specifically in relation to thermal destruction and thermo-oxidation processes.

b) At higher temperatures, chain fragmentation in silicone rubber can be accompanied by oxidation processes, which are facilitated by the concomitant action of heat and the presence of air.

c) The thermal effect on silicone rubber can be either long-term or occasional, such as impulse-like. The superior thermal properties of silicones compared to other plastics have led to a growing interest in heat-resistant and frost-resistant materials<sup>[4,5]</sup>.

The development of thermal analysis methods and instruments has provided an opportunity to study the degradation processes caused by thermal effects in silicones. In recent decades, significant experience and numerous results have been obtained in this field, primarily focusing on dimethylpolysiloxanes (DMS), which are extensively used for technical purposes. Density analysis methods at constant temperatures in different environments have been widely employed to investigate the destructive processes and thermal oxidation reactions of various polymers and rubbers<sup>[6-8]</sup>. It has been demonstrated that the thermal degradation products of DMS include cyclic compounds such as D3, D4, and others.

Some researchers have conducted studies on the thermal degradation of dimethylpolysiloxanes (DMS) in a nitrogen environment. They discovered that the reaction rate reached its highest value at 443°C. Additionally, they found that the thermal stability of the polymer increased when OH-hydroxyl terminated groups in the polymer chain were replaced by trimethylsilyl (CH)<sub>3</sub>Si groups, without changing the volume of volatile substances. However, the presence of traces of potassium hydroxide (KOH) in the polymer enhanced its destruction, resulting in the release of a small amount of methane.

The objective of this study was to investigate the impact of vinyl groups on the thermomechanical properties and resistance of dimethylsilicone rubber to organic and inorganic solutions. The analysis of changes in specific gravity at specific temperatures has been utilized to study the stability of polymers and silicone rubber.

## 2. Methods and materials

### 2.1. Materials research

- Polydimethyl silicone with a viscosity of  $\eta = 1 \times 10^6$  cs.
- Polymethyl vinyl silicone with a viscosity of  $\eta = 50000$  cs and containing 2-3-5% vinyl groups, obtained from Finomvegyzerszövetkezet-Hungary.
- Fumed silica as a reinforced filler, with a particle size of 5-30 nm and a surface density of  $300 \pm 30$  g/cm<sup>2</sup>.
- Low viscosity liquid polydimethyl silicone with a viscosity of  $\eta = 10$  cs.

- 2,4-dichlorobenzoyl peroxide as a catalyst for the rubber vulcanization reaction.

## 2.2. Rubber samples preparation

The sample preparation process consisted of several stages:

- The batch preparation involved mixing dimethylsilicone and methylvinylsilicone polymer samples separately with fumed silica as a reinforcing substance and a reagent to prevent the formation of folds. This was done in a mechanical mixer at a temperature of 150°C. The polymers were mixed in weight proportions of 2-3-5% vinyl groups, with homogeneous mixtures obtained by adding portions with a 10-minute interval between them and other components<sup>[11-13]</sup>.
- Sample 1 was made of dimethyl silicone polymer, with a molecular formula of  $\text{CH}_3[\text{Si}(\text{CH}_3)_2\text{O}]_n\text{Si}(\text{CH}_3)_3$ .
- Sample 2 was made of methyl vinyl silicone polymer, with a molecular formula of  $\text{C}_{13}\text{H}_3\text{OO}_3\text{Si}_4$
- Sample 3 was made of dimethyl silicone rubber.
- Sample 4 was made of methylvinylsilicone rubber, containing 2% vinyl by weight.
- Sample 5 was made of methylvinylsilicone rubber, containing 3% vinyl by weight.
- Sample 6 was made of methylvinylsilicone rubber, containing 5% vinyl by weight.

## 2.3. Vulcanization

Compound 4, 2-dichlorobenzoyl peroxide, was added as a catalyst to the prepared samples. The samples were mixed using an internal mixer under a pressure of 16.2 mPa (160 atmospheres) and a temperature of 150°C. To complete the vulcanization process, the resulting sheets with a thickness of 2 mm were placed in an oven at a temperature of 150°C for four hours. All samples also contained 40% fumed silica, 20% anti-crease, and 1.5% catalyst. It is important to note that rubber vulcanization is typically performed either at room temperature (RTV) or at high temperature (HTV).

## 2.4. Measurement method

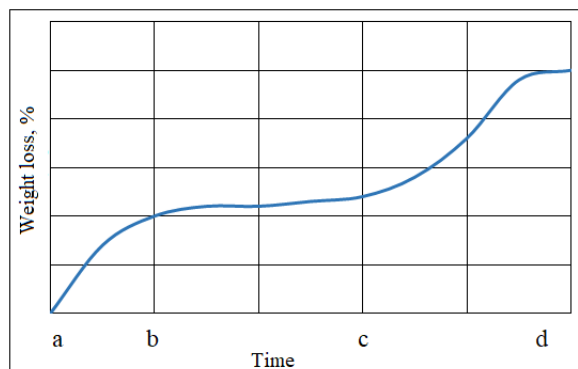
The weighing device is a rod with the sample on the lower porcelain part, which is placed inside the furnace at the operating temperatures  $\pm 0.2$  °C **Figure (1)**.



**Figure 1.** Thermo-furnace and weight-measuring device.

The weight loss of each sample was monitored for 24 hours at the specified temperature regime: 160, 180, 200, and 220°C. At ambient temperature, the weight of the polymer sample was measured to be  $1.000 \pm 0.0002\text{g}$ .

To eliminate any volatile substances that could impact its thermal properties, the rubber sample was subjected to a temperature of 200°C for four hours<sup>[14]</sup>. The graph in **Figure (2)** illustrates the weight loss of the test sample over time.



**Figure 2.** Weight change of silicone rubber over time at a constant temperature.

**Figure 2** shows three sections of the thermal analysis process:

a-b: Evaporation of volatile substances

b-c: Thermal degradation

c-d: Thermal degradation simultaneously with thermal oxidation, with thermal oxidation predominating.

It was observed that the evaporation of silicone polymer components is complete within 10-15 hours.

The process of thermal degradation can be described using the following mathematical equation<sup>[15]</sup>:

$$V = -\frac{dx}{dt} = K(1-x) \quad (1)$$

where:

$V$  - rate of degradation

$K$  - constant of the degradation process

$x$  - molar fraction of the degradation product

Given the small value of weight loss, some simplifications can be made in these calculations.

The overall reaction rate for the entire process can be determined using the following equation:

$$V = K(1-x) \quad (2)$$

The integration of the above equations can be expressed as:

$$\ln V = Kt(1-x) \quad (3)$$

From equation (3), the rate constant of thermal degradation ( $K$ ) can be calculated using the values from Table 1:

$$K = (x_2 - x_1) / (t_2 - t_1) \quad (4)$$

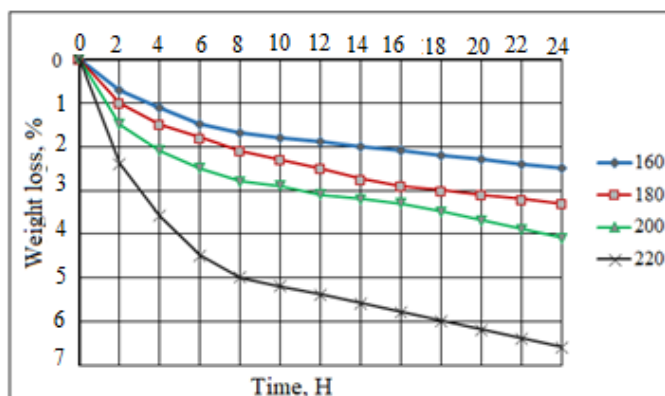
The half-life period of the polymer and rubber can be calculated using the rate constant of thermal

degradation ( $K$ ) as:

$$t_{1/2} = \ln 2 / K \quad (5)$$

### 3. Results and discussion

According to the test results conducted to measure the change in sample weight within a 24-hour period, graphs illustrating the relationship between weight reduction and time were constructed at four different temperature regimes: 160°C, 180°C, 200°C, and 220°C. **Figure 3** displays these graphs for sample 6 as an example.



**Figure 3.** Weight loss, expressed over time at temperatures 160, 180, 200, 220 °C.

The constants  $t_{1/2}$  and  $k$  can be calculated for the specified samples using ratios (1, 2).

**Table 1.** The values  $x$  at operating temperatures.

$$x = (x_2 - x_1)$$

$x_2 = 1.00 \text{ g - sample weight before drying,}$   
 $x_1$ - sample weight after drying  
 $t = t_2 - t_1 = 24 \text{ hours}$

Temperature	Polymer samples						Rubber samples					
	1		2		3		4		5		6	
	$x_1$	$x$	$x_1$	$x$	$x_1$	$x$	$x_1$	$x$	$x_1$	$x$	$x_1$	$x$
160 °C	.9916	.0084	.9782	.0217	.9932	.00672	.9962	.0037	.9943	.0056	.9925	.0074
180 °C	.98951	.01048	.9775	.0224	.9925	.0074	.995	.0044	.9916	.0084	.9907	.0092
200 °C	.98651	.01348	.9767	.0232	.9917	.0082	.994	.006	.9899	.0100	.9895	.0104
220 °C	.9689	.03100	.976	.024	.990	.009	.991	.009	.9844	.0156	.9835	.0164

**Table (2)** shows the values of the rate constant of the thermal degradation reaction  $k$  and the sample  $t_{1/2}$  half-life period for polymer and rubber samples at fixed temperature values.

According to **Table 2**, the rate constant values for the thermal degradation reaction of dimethylsilicon polymer (sample 1) clearly change at 220°C, with a value of 12.92 compared to methylvinylsilicon polymer (sample 2), which has a value of 10.0. This is because the process of thermal oxidation of the polymer may

occur simultaneously with the polymer destruction process.

**Table 2.** The values of the rate constant of the thermal degradation reaction  $k$  and the sample  $t_{1/2}$  half-life period.

Temperature	1 – $K \cdot 10^{-4}$ h 2- $t_{1/2}$	Polymer samples			Rubber samples		
		1	2	3	4	5	6
160 °C	1 – $K \cdot 10^{-4}$ h	3.5	9.06	2.80	1.56	2.36	3.12
	2- $t_{1/2}$	1972	761	2484	4423	2331	2211
180 °C	1 – $K \cdot 10^{-4}$ h	4.37	9.37	3.12	1.87	3.50	3.87
	2- $t_{1/2}$	1577	736	2211	3689	1971	1782
200 °C	1 – $K \cdot 10^{-4}$ h	5.62	9.68	3.43	2.50	4.20	4.37
	2- $t_{1/2}$	1227	712	2011	2760	1642	1578
220 °C	1 – $K \cdot 10^{-4}$ h	12.92	10.0	4.06	3.75	6.50	6.87
	2- $t_{1/2}$	534	690	1699	1840	1061	1001

Comparing the values for the two samples (1, 2), it is noted that the highest value is observed in sample 2 at temperatures of 160°C, 180°C, and 200°C, indicating that the disintegration of the methylvinyl silicon polymer is higher than that of the dimethylsilicon polymer. This is due to the fact that the chain length of the methylvinyl silicone polymer is shorter than that of the dimethyl silicon polymer, which is directly related to the breakage of the polymer chain.

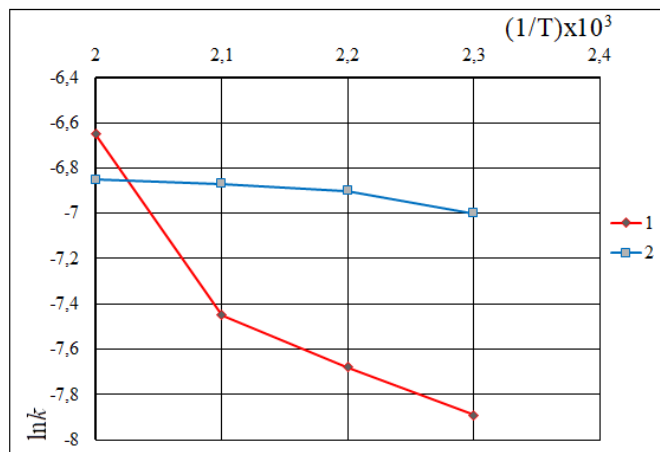
**Table 2** demonstrates that as the temperature increases, the values of  $K$  increase and the values of  $t_{1/2}$  decrease. This trend accurately reflects the reality and allows for a comparison of the properties of dimethylsilicon and methylvinylsilicon polymers when substituting a methyl group in the polymer chain with a vinyl group.

Upon analyzing the values of  $K$  for samples 1 and 3, it is evident that for sample 3 at a temperature of 220°C, the value is smaller compared to sample 1. This suggests that there is no thermal oxidation of the polymer in sample 3. This can be attributed to the formation of a bond between the hardener and the polymer chain, which hinders the oxidation process of the polymer.

When examining samples 4, 5, and 6, it is observed that in samples 5 and 6, the values of  $K$  increase by two or more times compared to sample 4. This indicates that the rate of degradation in samples 5 and 6 is higher. Based on this, it can be concluded that sample 4, made of methylvinylsiloxane rubber with a vinyl group content of 2% by weight, is the most favorable.

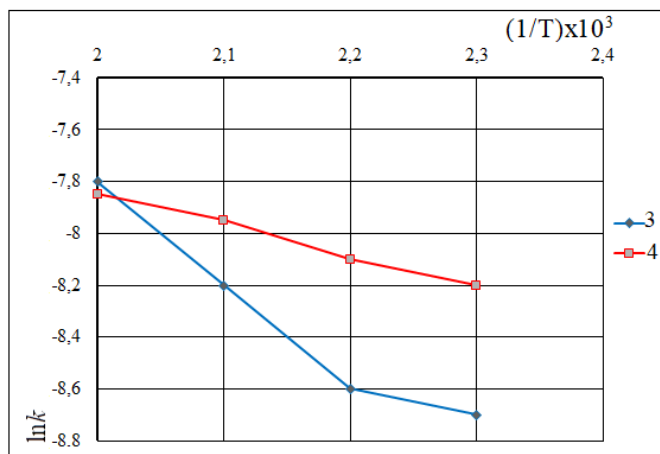
By utilizing the values of  $K$  at different temperatures, it is possible to generate a graphical curve of  $\ln(K)$  as a function of  $1/T$  for polymer samples 1 and 2. The data points for sample 1 do not align on a straight line, unlike sample 2, where all the points lie on a straight line (particularly at a temperature of 220°C). This confirms that the vinyl group,  $\text{CH}_2\text{-CH}$ , reduces polymer degradation and enhances its thermal oxidation compared to the presence of the methyl group,  $\text{CH}_3$ , at the same temperature because the molecular weight of dimethyl silicone rubber is generally higher than that of methyl vinyl siloxane rubber. Dimethyl silicone rubber typically has a higher molecular weight due to the presence of two methyl ( $\text{CH}_3$ ) groups on each silicon atom,

whereas methyl vinyl siloxane rubber contains one methyl group and one vinyl ( $\text{CH}_2=\text{CH}-$ ) group on each silicon atom. This correlation is depicted in **Figure 4**.

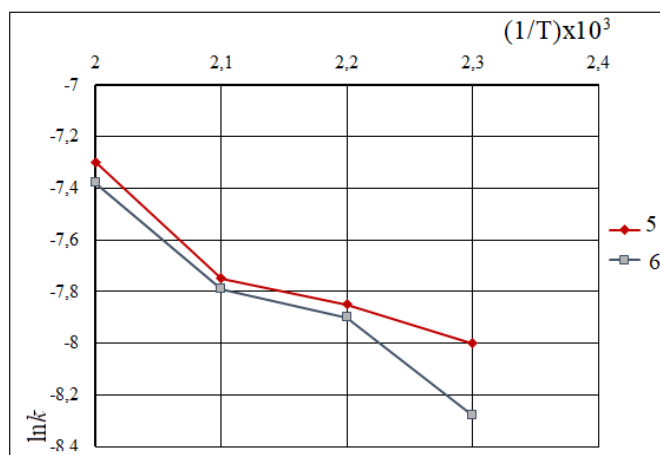


**Figure 4.** Curves of dependences  $\ln k$  on  $1/T$  for polymer samples 1 and 2.

The curves of dependences ( $\ln K$ ) on  $1/T$  for rubber samples 3 and 4 are shown in **Figure 5**, and for samples 5 and 6-in **Figure 6**.



**Figure 5.** Curves of dependences ( $\ln K$ ) on  $1/T$  for rubber samples 3 and 4.



**Figure 6.** Curves of dependences ( $\ln K$ ) on  $1/T$  for rubber samples 5 and 6.

By comparing the graphical curves of samples 4, 5, and 6, we observe that not all points are located on

the same straight line in the cases of samples 5 and 6, especially sample 6. This indicates the presence of thermal oxidation process in addition to thermal destruction process. However, in sample 4, it can be noticed that the points are located on a straight line, confirming that the material structure from which it is made helps to restrain the degradation of rubber and reduce the speed of this process. This is consistent with the measurements  $K$  and  $t_{1/2}$ , confirming that the presence of vinyl group in rubber with a content of 2% vinyl by weight (sample 4) increases the service life of methyl silicone rubber.

Based on the above, it can be stated that this method of studying changes in polymer and rubber weight at a constant temperature for a certain period of time allows us to determine more than one constant simultaneously and observe multiple processes to which polymers and silicone rubber are exposed (destruction and oxidation) through the measured constant values.

### 3.1. Thermodynamic constants

The values of the thermal destruction constant  $k$  calculated due to the results of changes in the polymers weight at the constant temperatures allow us to determine the paroxysmal reaction of thermal destruction  $\Delta H^*$  of both polymer and rubber based on the Arrhenius equation:

$$K = A.e^{-\Delta H^*/RT} \quad (6)$$

Making  $k$  depending on  $1/T$ , a straight line with a slope  $(-\Delta H^*/R)$  is obtained, and the entropy of activation  $\Delta S^*$ , taking into account  $\Delta H^*$ , can be calculated by the ratio [15]:

$$\Delta S^* = 19.150 \times \left( \frac{\log K + \Delta H^*}{19.10T} - \log 2.08 \times 10^{10} T \right) \quad (7)$$

Taking into account  $\Delta H^*$  and  $\Delta S^*$ ,  $\Delta G^*$  can be calculated by the following ratio:

$$\Delta G^* = \Delta H^* - T.\Delta S^* \quad (8)$$

**Table 4** also shows the values  $\Delta H^*$ ,  $\Delta S^*$  and  $\Delta G^*$  for the studied samples.

**Table 4.** The values  $\Delta H^*$ ,  $\Delta S^*$  and  $\Delta G^*$  for the studied samples.

Samples	$\Delta H^*$ , kJ/mol	$\Delta S^*$ , kJ/mol	$\Delta G^*$ , kJ/mol
Dimethyl silicon polymer	20	-0.288	155
Methyl vinyl silicon polymer	21	-0.321	153
Dimethyl silicone rubber	10	-0.298	150
Methylvinylsiloxane rubber containing 2 % of vinyl by its weight	22	-0.293	158
Methylvinylsiloxane rubber containing 3 % of vinyl by its weight	19	-0.292	156
Methylvinylsiloxane rubber containing 5 % of vinyl by its weight	20	-0.291	156

**Table 4** demonstrates that sample 4 exhibits the highest value of  $\Delta H^*$  when a vinyl group is present in



rubber with a 2% vinyl content by weight. This can be attributed to the potential formation of relatively stable intermediates during the degradation process, as compared to samples 5 and 6. Additionally, all studied samples display positive values of  $\Delta G^*$ , indicating the presence of a quasi-stable state at any given temperature.

### 3.2. Mechanical properties

The mechanical properties of vulcanized sheets were evaluated for samples of methylsilicon and methylvinylsilicon rubber with respective vinyl contents of 2%, 3%, and 5%. Tensile strength, tear strength, and elongation were measured using a ZMGI-200 device at a stretching speed of 250 mm/min, while hardness was assessed using a Shore A device. The mechanical properties of the different rubber samples are presented in **Table 5**.

**Table 5.** The mechanical properties of various rubber samples.

Mechanical properties of rubber samples after being exposed to a temperature of 200 °C for 24 hours	Dimethyl silicone rubber	Methylvinylsiloxane rubber containing 2 % of vinyl by its weight	Methylvinylsiloxane rubber containing 3 % of vinyl by its weight	Methylvinylsiloxane rubber containing 5 % of vinyl by its weight
Tensile Strength, $N/mm^2$	8.06	5.58	5.25	4.21
Tear Strength, N/mm	35	31	24	15.5
Elongation %	380	352	271	190
Hardness Shore A	56	53	55	56

Each of the values presented in the table represents the average value of five dimensions.

The analysis of the results in **Table 5** indicates that pure dimethylsilicon rubber possesses favorable mechanical properties due to its high molecular weight and strong bond with fumed silica filler particles. However, these mechanical properties decrease linearly with an increase in the percentage of vinyl in the rubber. In particular, sample 4 with 4% vinyl weight shows a smaller decrease compared to samples 5 and 6. This decrease can be attributed to the increased cross-linking that likely occurs as a result of the presence of vinyl groups. Vinyl groups have the capability to undergo reactions that lead to the formation of additional cross-links between the polymer chains. These additional cross-links contribute to the enhancement of mechanical properties and improved resistance to solvents observed in sample 4. The increased cross-linking also helps stabilize the rubber material and reduce the rate of rubber chain bond rupture during degradation processes.

As a result, sample 4 exhibits superior improvements in rubber properties compared to the other samples. On the other hand, in samples 5 and 6 where the vinyl percentage is higher, the mixture becomes more disordered, and the absence of vinyl in the cross-linking sites negatively impacts the mechanical properties of the resulting rubber, as indicated in the table.

The addition of vinyl groups to the rubber polymer chain affects its properties. In general, increasing the vinyl content in the rubber increases the crosslinking density, which leads to a higher Shore A hardness and lower elongation.

In the case of Sample 4Methylvinylsiloxane rubber containing 2% vinyl, it has a lower vinyl content compared to Samples 5Methylvinylsiloxane rubber containing 3% vinyl and 6Methylvinylsiloxane rubber containing 5% vinyl. This means that Sample 4Methylvinylsiloxane has a lower crosslinking density, resulting in greater elongation and lower Shore A hardness.

### 3.3. Solvent resistance

The resistance of various rubber samples to organic and inorganic solvents was evaluated using the swell-weight equilibrium method. Small pieces of rubber samples were placed in specific solvents at room temperature and weighed before and after one week of exposure. The difference in weight was calculated to determine the extent of swelling caused by each solvent. Table 6 presents the effect of different solvents on the rubber samples.

**Table 6.** The effect of various solvents on rubber samples.

Solvent type, 7 days at room temperature	Dimethyl silicone rubber	Methylvinylsiloxane	Methylvinylsiloxane	Methylvinylsiloxane
		rubber containing 2 % of vinyl by its weight	rubber containing 3 % of vinyl by its weight	rubber containing 5 % of vinyl by its weight
(Weight Change) %				
10% $H_2SO_4$	+0.65	+0.28	+0.35	+0.56
10% $HCL$	+2	+0.056	+0.072	+0.30
$CH_3COOH$ <i>Acetic acid</i>	+3	+4.07	+4.65	+5.07
$NH_4OH$	+2	+1.30	+1.38	1.48
Acetone	+15	+7.42	+8.34	+12.21
Isopropyl	+10.3	+6.96	+6.41	+6.07
Methanol	+3.2	+1.85	+1.95	+2.2
Hexane	+180	+168	+157	+139
Itra	+220	+213	+191	+167
Vegetable oil	+0.95	+0.38	+0.55	+0.84

The results presented in **Table 4** demonstrate that sample 4, composed of methylvinylsilicon rubber, exhibits superior resistance to solvents and oil compared to samples 5 and 6. Therefore, it can be concluded that the optimal percentage of vinyl groups in the rubber is 2% by weight.

The sample 4 has a lower crosslinking density compared to Samples 5 and 6. A lower crosslinking density means that the rubber chains in Sample 4 allow solvents to penetrate and swell the rubber more easily. This leads to higher swelling in hexane and Itra.

On the other hand, Samples 5 and 6 have higher crosslinking densities due to their higher vinyl content. The increased crosslinking density restricts the penetration of solvents into the rubber matrix, resulting in lower swelling in hexane and Itra.

## 4. Conclusions

To study polymer and silicone rubber samples, we employed the weight change analysis method and the swell-weight equilibrium method at constant temperatures over an extended period of time. These methods

allowed us to assess the stability of these materials for potential industrial applications. Furthermore, they enabled us to observe multiple processes simultaneously, including degradation and thermal oxidation.

Upon analyzing the results, it became evident that sample 4, composed of methylvinylsilicon rubber with a vinyl group content of 2% by weight, exhibited superior improvements in rubber properties. Specifically, this sample demonstrated a reduced rupture rate of rubber chain bonds, enhanced resistance to various solvents, improved mechanical properties, and more. Collectively, these findings contribute to an increased service life of the rubber material.

## Conflict of interest

The authors declare no conflict of interest.

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