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Performance of PVC/SiO₂ nanocomposites under thermal ageing

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Abstract

Accelerated thermal ageing test on nanocomposites of polyvinyl chloride (PVC/SiO₂) is conducted at constant temperatures of 110 °C and 140 °C. Accordingly, different PVC/SiO₂ nanocomposite samples with different silicon dioxide (SiO₂) filler loading are prepared. The preparation of the samples was carried out based on the solution casting technique. The loading concentrations of SiO₂ in the investigating samples are 0, 1, 2.5, 5 and 7.5 wt%. The breakdown strength of the prepared PVC/SiO₂ nanocomposite samples is evaluated before and after the thermal ageing test. The evaluation of breakdown strength is carried out based on American Society for Testing and Materials (ASTM) standard. Also, dielectric spectroscopy, which includes dielectric constant (ϵ) and tangent loss (tan δ) of nanocomposite, is performed before and after the thermal ageing test. Also, the mass losses during thermal ageing of all samples are evaluated. The results show that the breakdown strength, tan δ , and the dielectric constant (ϵ) of pure PVC and PVC/SiO₂ nanocomposites are significantly affected by the thermal ageing test. Adding a small amount of SiO₂ nanoparticles to PVC leads to improvement in its dielectric properties before and after the thermal ageing is less than that of pure PVC and confirms the obtained result from breakdown strength.

Keywords Polyvinyl chloride · Silicon dioxide · Nanocomposites · Dielectric properties · Mass losses · Thermal ageing

Introduction

Electrical cables are an important part in electrical transmission and distribution networks. It is covered with a suitable insulating material, based on the operating voltage level. When increasing the operating voltage level, it is necessary to increase the thickness of the insulation material which leads to an increase in the cost.

Polyvinyl chloride (PVC) is used as an insulating material in power cable construction. This is due to its good features such as easy processing, low cost, excellent mechanical properties, having greater rigidity than other

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general-purpose thermoplastic materials and good insulation performance (Shwehdi et al. 2003; Sugumaran 2015). PVC works safely at 75 °C in wet weather and at 90 °C in dry weather (CHEMinfo Services Inc 1997). Under overload or short circuit conditions, PVC-insulated cables are exposed to thermal stress (thermal ageing). This stress leads to gradual degradation of the initial product characteristics. The degradation causes irrevocable changes in the insulation properties, which rapidly reduce their lifetime. Therefore, the development of insulated cable is required to ensure long life under high temperatures (Shwehdi et al. 2003).

Nanocomposites have been proven to be able to improve the mechanical and electrical insulation materials of the polymer matrix (Sugumaran 2015, 2013; Yun et al. 2007; Tagami et al. 2008; Tanaka et al. 2005a, b; Green et al. 2008; Selvaraj et al. 2015; Mansour et al. 2016; Thenthiruppathi et al. 2014; Shah et al. 2009; Danikas and Tanaka 2009; Thomas 2009; Roy et al. 2005; Huang et al. 2014). However, under high temperature during the operation of polymers, the properties of nanocomposites during thermal ageing may be affected. Therefore, it is necessary to test nanocomposite materials in the long term before using in practical field.



A number of researches have evaluated unfilled polymer materials under thermal ageing stress (Shwehdi et al. 2003; Nedjar et al. 2006, 2003; Nedjar 2009; Mecheri et al. 2013; Jakubowicz et al. 1999; Royaux et al. 2017). However, very few researches have evaluated polymer nanocomposites under thermal ageing stress (Wang et al. 2017; Harvanek et al. 2016). In Wang et al. (2017), the insulation properties of low-density polyethylene (LDPE) after exposure to thermal ageing can be enhanced by adding SiO₂ as well as MgO nanoparticles (NPs). In another study (Harvanek et al. 2016), bisphenol A diglycidyl ether (DGEBA)-type epoxy resin filled with SiO₂ NPs was exposed to long-term thermal ageing as well as electrothermal effect. The aged samples' permittivity was found to be greater than that of the unaged samples. Also, during ageing, the loss tangent was increased. Moreover, the studies of the nanocomposites under thermal ageing (Wang et al. 2017; Harvanek et al. 2016) were limited to only a NPs single concentration of 1 wt%. The thermal ageing studies were not deeply discussed. Also, until now, no study has evaluated the dielectric properties of PVC nanocomposites under thermal ageing. So, the main objective of this work was to deeply investigate and discuss the thermal ageing effect on the dielectric properties of PVC/SiO₂ nanocomposites, considering different concentration levels of SiO₂ NPs at two different thermal ageing temperatures.

In this work, the effect of a concentration level of SiO_2 NPs on the dielectric properties of the PVC polymer during thermal ageing is evaluated. PVC nanocomposites considering different SiO_2 NPs concentration levels (0, 1, 2.5, 5 and 7.5 wt%) are prepared. Accelerated thermal ageing test is performed at two different temperatures, 110 °C and 140 °C. The evaluation of the breakdown strength of the prepared samples before and after the thermal ageing test is carried out based on the ASTM standard. The dielectric spectroscopy of all samples is measured before and after the thermal ageing test. Furthermore, the mass loss of samples is also evaluated during thermal ageing. This study gives some recommendations to increase the lifetime of power cables.

Experimental work conditions

In this section, the preparation procedure of PVC/SiO_2 nanocomposite samples is presented. Also, the breakdown strength test and dielectric spectroscopy measurements are reported. Finaly, the mass loss measuremet of the samples is presented.

Sample preparation

PVC granules used in this study were provided by Egyplast—Egyptian company for plastic industry (Elsewedy). These granules have a density of 1.51 g/cm³, a tensile



strength of 13 Mpa, and volume resistivity of 10¹³ ohm. cm at 23 °C. SiO₂ NPs were obtained from Sigma-Aldrich Company. SiO₂ NPs have a particle size of 20 nm and melting point greater than 1600 °C. Figure 1 shows the preparation precedure for PVC/SiO₂ nanocomposite samples. The required amount of PVC and the SiO₂ NPs for each sample were in total 3 g. Firstly, 40 mL of tetrahydrofuran (THF) was used to dissolve the required amount of PVC for each sample under stirring for 30 min at room temperature. Then, an amount of SiO₂ NPs (0,1, 2.5, 5 and 7.5 wt%) was directly added to the solution under stirring for another 90 min. To improve the NPs dispersion, the mixture was subjected to ultrasonic waves for 15 min using a sonicator. To avoid a rise in temperature of the solution during the sonication process, the mixture was put in a glass vessel inside a water bath. It was found that the solution temperature did not exceed 45 °C. This temperature is much less than the melting temperature of PVC. Then the mixture was poured into a Petri dish and left for 24 h at room temperature. The average thickness of the prepared samples was around 0.3 ± 0.02 mm. To ensure the extraction of THF solvent, the samples were left for 20 days in a vacuum chamber. To obtain the same conditions, the preparation procedure for the pure PVC sample was the same as in the PVC nanocomposite samples. Finally, the prepared pure and PVC nanocomposite samples were subjected to thermal ageing with temperatures of 110 °C and 140 °C.

In this work, field-emission scanning electron microscope (FE-SEM, Quanta FEJ20) is used to investigate the morphologies of PVC/SiO₂ nanocomposites and distribution of NPs in the polymer matrix.



Fig. 1 Preparation procedures for the PVC/SiO_2 nanocomposite samples

Breakdown strength test

The breakdown strength measurement is an essential test used to diagnose the insulating materials' dielectric state. The breakdown strength was measured according to the ASTM standard. The applied AC voltage, 50 Hz, was adjusted at a rate of 500 V/s. The breakdown strength test was carried out using a mushroom electrode type as shown in Fig. 2. The sample as well as the two electrodes was completely immersed in transformer oil to prevent surface flashover. For each sample, five times breakdowns were performed. The breakdown strength of each sample was computed by dividing the obtained breakdown voltage on the sample thickness. The evaluation of breakdown strength was carried out considering the average of these five breakdown strength values as recommended in different researches such as in Sugumaran (2013, 2015), Tanaka et al. (2005a, b), Mansour et al. (2016), Shah et al. (2009), and Harvanek et al. (2016). The breakdown strength test was carried out on all samples each week during the thermal ageing test at 110 °C. Howerver, in the case of 140 °C, the breakdown strength test was carried out every 3 or 4 days. All breakdown strength tests were performed on fresh as well as the aged samples at room temperature.

Dielectric spectroscopy measurements

Permittivity represents the ability of the dielectric material to allow its chain order with external electric field direction. Electrical losses possible in an insulating material which lead to increase in temperature can be expressed by the tangent loss (tan δ). The increase in tan δ is the indicator of the deterioration of the polymer. The dielectric constant ($\hat{\boldsymbol{\epsilon}}$) and tangent loss (tan δ) of the prepared samples were measured before and after the thermal ageing at various frequencies up to 1 MHz at an applied voltage of 1 V. In each sample, 15 times $\hat{\boldsymbol{\epsilon}}$ and tan δ were measured at each frequency



Fig. 2 AC breakdown test cell with VDE electrodes

using LABVIEW. Then the average value was obtained. The measurement was carried out using accurate HIOKI 3532-50 LCR Hi-tester device.

Mass loss test

Mass loss is a physical property which is used to estimate the degradation of the insulation. The weight of the prepared sample is measured before and after thermal ageing using a sensitive balance. This sensitive balance has a readout of 0.01 mg. The mass loss percentage during thermal ageing was estimated based on Eq. (1):

Mass loss % =
$$\frac{m_{\rm o} - m_{\rm i}}{m_{\rm o}} \times 100,$$
 (1)

where m_0 and m_i are the mass of the sample before and after thermal ageing, respectively.

Results and discussions

In this section, the evaluation of the prepared samples, including pure PVC and PVC/SiO₂ nanocomposites, is presented. The evaluation is carried out based on the measurement of breakdown strength and dielectric sprectroscopy. All these tests are performed on the fresh samples without any thermal ageing. Finally, the tests are re-evaluated during thermal ageing at 110 °C and 140 °C.

Evaluation of PVC/ SiO₂ nanocomposites

Here, the evaluation of pure PVC and PVC/SiO₂ nanocomposites considering the fresh samples without any thermal ageing is presented.

Dielectric strength

Figure 3 shows the effect of SiO₂ NPs concentration levels on the dielectric strength of the PVC samples. Generally, Fig. 3 reflects that there are enhancements in the breakdown strength by adding SiO₂ NPs to the PVC materials. The maximum enhancement percentage in breakdown strength is 7% for nanocomposite sample with concentration levels of 5% SiO₂ NPs as compared to the pure PVC sample. Then, the breakdown strength is decreased with increase in SiO₂ NPs concentration levels. The enhancment in breakdown strength is due to charge trapping action at the interface between NPs and the polymer matrix (Shah et al. 2009). The high surface area to volume of NPs in the polymer matrix leads to enhancment in the probability of charge trapping action. In addition to the presence of NPs act as a dispersion of the applied electric field and increase the path length necessary for the occurrence of electric breakdown (Danikas





Fig. 3 Variation of the dielectric strength versus filler concentration of the PVC/SiO_2 nanocomposite

and Tanaka 2009). In contrast, the decrease in breakdown strength at high concentration levels may be due to the increase in the probability of particle agglomeration as well as the increase of NPs' overlap probabilities. To confirm the obtained results, the particle distribution in PVC sample is examined by FE-SEM. Figure 4 shows the FE-SEM for PVC/SiO₂ nanocomposite considering low and high concentration level, 2.5 wt% and 7.5 wt%, respectively. At the low concentration level of 2.5 wt%, a homogeneous distribution of NPs and no agglomerations is illustrated as in Fig. 4.a. However, when the concentration level is increased to 7.5 wt%, an agglomeration of NPS in some region is seen, as shown in Fig. 4.b. Therefore, the decrease in breakdown strength at high concentration levels is due to NPs agglomerations.

Dielectric spectroscopy

The dielectric response of the prepared samples (pure PVC, PVC/SiO₂ nancomposites) is determined considering



Fig. 4 FE-SEM micrograph for nanocomposites with two magnifications (I) $3000 \times$ magnification and 40 µm scale, (II) $12000 \times$ magnification and 10 µm scale, **a** PVC/2.5 wt% SiO₂, **b** PVC/7.5 wt% SiO₂





Fig. 5 Variation of ϵ versus filler concentration of PVC/SiO₂ nano-composite

dielectric constant and tano. Accordingly, Fig. 5 illustrates the effect of SiO₂ NPs concentration levels on dielectric constant of PVC materials. It is shown that the dielectric constant of nanocomposite decreases with the increase in SiO_2 NPs concentration levels up to 2.5%. Then the dielectric constant of PVC nanocomposite begins to increase at a concentration level greater than 2.5% of SiO₂ NPs. Therefore, adding SiO₂ NPs to the PVC material decreases or increases its dielectric constant. This depends on the filler permittivity and the restriction of molecular movements in the interaction zone of NPs (Mansour et al. 2016). In the case of concentration levels lower than 2.5%, the restriction of molecular movements is dominant. More specifically, a greater number of interaction zones between NPs and polymer are created at low concentrations of NPs, due to the homogenous distribution of NPs, as declared in the previous section. This leads to hindrances in the mobility of the polymer chain as well as decreases the dielectric constant. In contrast, the increase of dielectric constant in the concentration levels of SiO₂ NPs greater than 2.5% is due to the effect of filler permittivity, where the dielectric constant of SiO_2 NPs is greater than that of the base PVC material. The effect of the dielectric constant for NPs is apparent at high concentration, due to some agglomeration as discussed in the previous section.

Figure 6 shows the variation of tan δ as a function of SiO₂ NPs concentration levels. It is clear that adding SiO₂ NPs to the PVC material can significantly decreases tan δ . Then tan δ slightly increased at 7.5% SiO₂ NPs concentration level. tan δ is mainly dependent on the material's electrical conductivity. The decrease in the electrical conductivity with SiO₂ NPs concentration up to 5% can be attributed to hindrances in charge transport through different chains and interfaces. In case of PVC nanocomposites with concentration level greater than 5%, the electrical conductivity increases. This is due to the increase in the agglomeration probability.



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Fig. 6 Variation of tan δ versus filler concentration of PVC/SiO₂ nanocomposite

From the aforementioned discussion, adding small amounts of SiO₂ NPs can improve the dielectric strength of PVC polymer materials. Also, the addition of SiO₂ NPs decreases the dielectric constant and tan δ of the PVC materials. So, adding small amounts of SiO₂ NPs to the base PVC materials can improve its electrical properties. This can be very useful in the power cable application without any ageing. Practically, the loaded cables suffer from thermal stress as well as thermal ageing. Therfore, to simulate the real field, the evaluation of PVC/SiO₂ nanocomposites should be carried out considering thermal ageing. This evaluation is performed as follows.

Evaluation of PVC/SiO₂ nanocomposites under thermal ageing

The performance evaluation of PVC/SiO_2 nanocomposites under thermal ageing is presented. The evaluation is carried out considering the pure PVC as well as PVC/SiO_2 nanocomposites at two thermal ageing temperatures, 110 °C and 140 °C.

Dielectric strength

Figure 7 shows the effect of ageing time on the breakdown strength of PVC/SiO₂ nanocomposite samples with different concentrations of SiO₂ NPs at 110 °C ageing temperature. The figure shows that the breakdown strength of the pure PVC and PVC/SiO₂ nanocomposite increases with increase in ageing time up to 40 days. Then, the breakdown strength is slightly decreased with increase in ageing time up to 80 days and after that a sharp decline occurs. The enhancement precentage in breakdown strength, obtained after 40 days of thermal ageing, is 30% at a concentration level of 1% SiO₂ NPs. The increase in breakdown strength for the first





Fig. 7 Variations in dielectric strength for PVC/SiO_2 nanocomposites with ageing time at 110 $^\circ\rm C$

interval ageing time can be attributed to loss of a part of the plasticizer and the arrangement of interlacing lines (Nedjar et al. 2006). In other words, thermal ageing for pure PVC causes some defects on the surface of the sample. The free charges from the electrode are located in these defects near the electrode. The accumulated charges in the defects are called homocharge, which increases breakdown strength. For the nanocomposite, the mobility of free charges increases after thermal ageing. The free charges are trapped in a bond and bounded region around the nanoparticles. The created homocharge in nanocomposite is more deeply from the sample surface. So the breakdown strength of the nanocomposite is dramatically increased by the thermal ageing effect. For ageing time beyond 40 days, the decrease in breakdown strength is caused by weakening molecular bonds, break of C-Cl bonds and the disappearance of the plasticizer.

To study the effect of ageing temperature on the breakdown strength PVC/SiO₂ nanocomposites, the evaluation is repeated at an ageing temperature of 140 °C. Figure 8 shows the effect of ageing time on the breakdown strength of PVC/ SiO₂ nanocomposite at 140 °C. From this figure, it can be seen that the breakdown strength is significantly decreased with increase in ageing time. At high ageing temperature, the rapid decrease in breakdown strength is due to the rapid loss of plasticizer (Nedjar et al. 2006; Nedjar 2009; Mecheri et al. 2013). At ageing temperature 140 °C there is no clear trend for the effect of SiO₂ NPs. This can be attributed to the fact that the PVC raw begins to decompose at high temperature.

Dielectric spectroscopy

Figure 9 shows the effect of ageing time on the dielectric constant of PVC/SiO_2 nanocomposite materials with different concentrations of SiO_2 NPs at ageing temperature 110 °C. This figure shows that the dielectric constant for





Fig. 8 Variations of dielectric strength for PVC/SiO₂ nanocomposites with the ageing time at 140 $^{\circ}$ C

both pure sample and nanocomposite with low concentration (1% SiO₂) initially slightly decreases for ageing time up to 80 days. Then it gradually increases. However, the dielectric constant of nanocomposite with high concentration of SiO₂ NPs either increases or seems to be constant. The decrease in the dielectric constant can be attributed to rearrangement of molecules. The variation of the dielectric constant as a function of SiO₂ NPs at different ageing times is shown in Fig. 10. This figure reflects that the dielectric constant of nanocomposite with low concentration level is lower than that obtained for pure PVC before ageing, while the dielectric constant of all nanocomposites is higher than that obtained for PVC after beginning ageing. After beginning ageing, the interaction between filler and polymer for all concentrations is decreased. Therefore, the molecular bonds are weakened and the free volume is raised. So the dielectric constant of the filler is dominant. It can be seen



Fig. 9 Variations of ϵ for PVC/SiO₂ nanocomposites with the ageing time at 110 °C and frequency 1 MHz



Fig. 10 Variation of ϵ versus filler concentration of PVC/SiO₂ nanocomposite at 110 °C



Fig. 11 Variations of ϵ for PVC/SiO₂ nanocomposites with the ageing time at 140 °C and frequency 1MHZ

from Fig. 11 that the dielectric constant is increased with ageing time at ageing temperature 140 °C. For example, the dielectric constant of nanocomposites with 2.5% from SiO₂ NPs is increased from 4.77 before ageing to 8 after ageing. The reason is that the increasing in ageing time leads to a reduction in viscosity. The reduction in viscosity enhances the mobility of the polymer chains and enhances the molecular polarization (Nedjar et al. 2006).

The effect of ageing time on tan δ for PVC/SiO₂ nanocomposite with various concentrations of SiO₂ NPs at ageing temperature 110 °C is shown in Fig. 12. It can be seen that the variation of tan δ for pure PVC is not significant at ageing temperature 110 °C up to 75 days. Then beyond this interval, tan δ is sharply increased. In case of the PVC/SiO₂ nanocomposite, the variation of tan δ for nanocomposite is not significant at ageing temperature 110 °C up to 100 days. Then beyond this interval, tan δ is gradually increased.



80

100

120

140

0.20

0.15

ທີ່ ເສັ 0.10

0.05

0.00

20

40

Fig. 12 Variations of tan δ for PVC/SiO₂ nanocomposites with the ageing time at 110 °C and frequency 1 MHz

Ageing Time (days)

60



Fig. 13 Variation of tan δ versus filler concentration of PVC/SiO₂ nanocomposite at 110 °C after 125 days

The variation of tan δ for PVC as a function of SiO₂ NPs after 125 days of thermal ageing at 110 °C is shown in Fig. 13. It is clear that $\tan \delta$ for pure PVC is higher than that obtained for PVC/ SiO₂ nanocomposite. tan δ values are mainely related to electrical conductivity. The increase in electrical conductivity with ageing time can be attributed to increase in free volume as well as weak bond. The degradation of PVC/ SiO₂ nanocomposite samples occurs after a longer period of thermal effect as compared with the pure PVC sample. According to the Tanaka model (Tanaka et al. 2005), the interface between nanoparticle and polymer can be divided into three parts: bond layer, bounded layer, and loose layer. The stabilization of the nanocomposite samples, which means restriction of polymer chains, may be attributed to high coupling between NPs and polymer chain. Therfore, the mobilities of chains as well as $\tan \delta$ in PVC/SiO₂ nanocomposites are smaller than that obtained in pure PVC.



Figure 14 shows the effect of ageing time on tan δ for pure as well as PVC nanocomposite at ageing temperature 140 °C. The results show that tan δ remains constant for ageing time up to 15 days. Then tan δ was increased with increasing of ageing time. At ageing temperature 140 °C, there is no clear trend for the effect of SiO₂ NPs on tan δ due to the same reason as discussed in the previous section.

Mass loss

The results of mass loss for pure PVC as well as PVC/ SiO₂ nanocomposite samples as a function of ageing time at 110 °C and 140 °C are illustrated in Figs. 15, 16, respectively. At ageing temperature 110 °C, the percentage of mass loss was increased by increasing the ageing time for the pure as well as PVC/SiO₂ nanocomposites samples as shown in Fig. 15. This can be attributed to the increase in of plasticizer loss with increase in ageing time (Royaux et al. 2017; Gonzalez et al. 2006). More specifically, the loss of plasticizer is done in two steps: the first is the spread of the plasticizer molecules from the place to the surface and the second phase evaporates the plasticizer from the surface. At thermal ageing temperature 140 °C, both of plasticizer loss and HCl loss occur very quickly (Nedjar et al. 2003; Jakubowicz et al. 1999; Royaux et al. 2017; Gonzalez et al. 2006), as illustrated in Fig. 16. The rate of mass loss at ageing temperature 140 °C is higher than that obtained at ageing temperature 110 °C. Mass loss for nanocomposite at each two temperature of thermal ageing is less than that obtained for pure PVC. Thermal stabilization and low mass loss for nanocomposite can be attributed to more interfaces between SiO₂ NPs and polymer chain, which restrict and inhibit emission of small parts during the thermal ageing effect.



Fig. 14 Variations of tan δ for PVC/ SiO₂ nanocomposites with the ageing time at 140 °C and frequency 1 MHz





Fig. 15 Variations of % weight losses for PVC/SiO₂ nanocomposites with the ageing time at 110 $^{\circ}$ C

Conclusion

In this paper, the evaluation of dielectric properties of PVC/ SiO₂ nanocomposites was carried out. Different nanocomposite samples were prepared considering different concentrations of SiO₂ NPs. The evaluation was carried out based on the measurement of breakdown strength, dielectric spectroscopy, and mass loss. All these properties were evaluated before and after thermal ageing at 110 °C and 140 °C. Based on the present study, the following points can be concluded:

1. Adding a small amount of SiO_2 NPs to the PVC polymeric material enhances its breakdown strength by 7% without ageing. However, the maximum percentage enhancement reached is about 30% during thermal ageing at 110 °C.



Fig. 16 Variations of % weight losses for PVC/ SiO_2 nanocomposites with the ageing time at 140 $^{\circ}\rm C$

- 2. The dielectric constant of PVC decreases by adding small amounts of SiO_2 NPs without ageing. However, the dielectric constant of both pure and PVC/ SiO_2 nanocomposites was enhanced during thermal ageing at 140 °C.
- 3. Adding SiO₂ NPs to PVC polymeric material decreases its tan δ without thermal ageing. On the other hand, thermal ageing increases tan δ for both pure and PVC/SiO₂ nanocomposite samples. However, increase of tan δ in the PVC/SiO₂ nanocomposite samples is less than that obtained for the pure PVC sample. Therefore, the antithermal ageing ability of PVC materials was enhanced by adding SiO₂ NPs.
- 4. The rate of mass loss in PVC/SiO₂ nanocomposite at two thermal ageing temperatures of 110 °C and 140 °C is less than that obtained for pure PVC. Therefore, the power cables with PVC/SiO₂ nanocomposite have a long lifetime compared to the cables with pure PVC.

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