



Novel Flame Retardant HDPE Corrugated Pipes as a Riser Protector in Agriculture

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ABSTRACT

In this article flame retardant HDPE corrugated pipes as a riser protector in agriculture were produced by adding 59% magnesium di-hydroxide additive to HDPE raw materials by using a single extruder. The compound was used for pipe production and the resulted pipe was compared with pristine pipe under OIT, DSC, MFI, Density and ash content experiments. The mechanical properties of the pipes were measured by ring stiffness, flexibility, falling weigh tests. Results indicated that the produced pipes are flame resistant and showed acceptable mechanical properties. All tests were done in reference to related standards.

Keywords: Flame Retardant, magnesium di-hydroxide, Corrugate Pipe, High-Density polyethylene, Riser Protector

1. INTRODUCTION

Nowadays polymer pipelines have extensive applications in agriculture. The most used polyethylene pipe in agriculture is single-layer pipes. However in our country the polyethylene corrugated pipes are used as a riser protector in agricultural lands. Risers have a significant role in irrigation but these items are not flexible though they are vulnerable, in order to abatement of fracture or separation from twisted areas, they should be equipped with a protector in order to prevent strokes from drifts [1, 2]. In the past, the riser protectors were made of concrete which was heavy and had outrageous production and transportation expenses. For ease of this application, PVC pipelines in diverse sizes were implemented but because of low strength and slight impact of these pipes, they were criticized. Currently in order to extend the fields and increase the project efficiency, polyethylene pipes are one of the top options. In the other side, single layer pipes are light weighted and this leads to abduction of these pipes in the fields. Hence since three years ago corrugated pipes were used for this purpose [3-6]. Because of below mentioned exclusivities, these pipes have preferences among other pipe groups:

- Corrugated pipes are weightless in comparison with concrete protectors and single layer pipes, and their portage is easier.
- Corrugated pipes unlike single layer pipes can be cut easily with cutter in the place of usage and do not need any professional cutting tool like saw.
- Corrugate pipes are cheaper in comparison with single layer pipes.
- These kinds of pipes owing to their jagged outer surface can be filled up with mud and soil and because of this fact, pipe abduction will be impossible.

For these reasons, corrugated pipes are a proper choice for this purpose. There is a major challenge in corrugated pipe usage in agriculture, after product harvesting, farmers burn their fields for elimination the leftovers and pugs. Due to the fact that polyethylene pipes can't resist fire, they will be burnt [1-3]. Polymers owing to their chemical structure which contain hydrogen and carbon have flame capacity. Fame reaction occurs because of these two reasons [4-6]:

- Presence of one or several agents with flame capability (reducer)
- Presence of one or several flame creator agents (oxidizer) which is almost the oxygen in air

Flame reactions almost initiate because of temperature increase leading to bond session. Volatile content from polymer decomposition emit in the air and a mixture of gasses with the flame ability (flammable gasses) is produced. By temperature increasing to the combustion temperature (i.e. the lowest temperature which a material without any external stimulant like spark or flame in ordinary atmosphere can catch fire on their own indeed the temperature in which flame reaction happens.), the combustion reaction happens and heat is produced [5-8].

1.1 Flame Retardant Material Mechanisms

1. Take participates in flame propagation mechanism chemically.
2. It is possible that the huge volume of inflammable gasses produce which can decrease oxygen consumption.



3. It is possible that decomposition by implementing endothermic reactions causes to absorption of heat.
4. By constructing an impenetrable coating toward flame, the oxygen entrance in the polymer is blocked [1, 4].
In other words, function of flame retardants has two features:

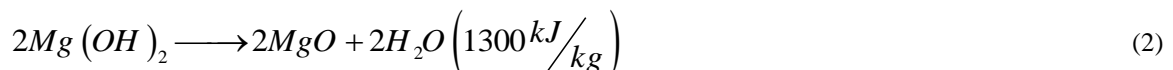
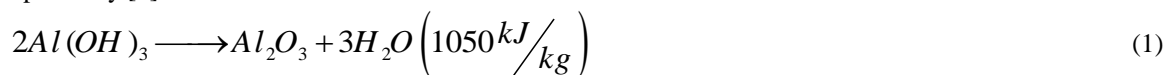
1.1.1 Physical Feature

Endothermic decomposition of some flame retardant materials leads to temperature diminution below the combustion temperature. In this case we can introduce hydrogen magnesium or hydrated tri-alumina which can decrease the temperature by steam exemption in 200 and 300 °C respectively. Another approach is dilution of gas phase in the surrounding of ignition system by non-effective gasses (like H₂O, CO₂, NH₃ and so on), which reduce the flame possibility and restricts the access of oxygen to fuel [9, 11].

1.1.2 Chemical feature

Exemption of free radicals (Cl or B) can cease the combustion in the gas phase. These radicals can react with active free radicals (hydrogen and hydroxyl) and produce compositions which have less reactivity or are non-effective mixtures. Modification of combustion process leads to decreasing the exothermic reactions and this is followed by temperature decrease of the system. In condense phase, chemical reactions can lead to accelerate of polymer chain rapture and after that the polymer droplets exit from flame area or by producing ashes on the surface of polymer, act as a physical insulator between gas and dense phase [10-13].

Selecting a flame retardant for a polymeric composite is performed on the basis of cost, chemical compatibility with polymer, heat decomposition temperature and weight. Many of the flame retardant fillers reduce the mechanical properties of composite and this subject is determinative in selecting a flame retardant. In some cases, flame retardant materials reduce the flame ability but increase the smock and toxic gas emotion from material decomposition. For this reason, implementation of flame retardant mixtures in order to minimize the side effects of these materials in mechanical properties and smock emission is controversial [11, 10, 14]. Two examples of commonly used flame retardant additives are Aluminum tri-hydroxide (ATH) and magnesium di-hydroxide (MDH). The two following equations show ATH and MDH decomposition respectively [3].



Decomposition temperature of ATH is between 180 and 200°C and according to its decomposition reaction negative effects in flame capacity reduction is created:

- Heat absorption cause polymer cooling.
- Produced Al₂O₃ act as a heat insulator.
- The product vapor causes dilution of flammable gasses and produces a protector gas layer.

Although due to the low decomposition temperature, this composition is used in some polymers, for polymers with high working temperature, usage of MDT with decomposition temperature more than 300°C is suggested [3, 15, and 16].

In this article, Polymer Ava Novin Iranian Company introduced a mineral additive based on magnesium di-hydroxide in corrugated pipelines for enhancement of their fire resistance property in order to use as a riser protector in agricultural lands.

2. EXPERIMENTAL

2.1 Materials

Industrial grade Magnesium peroxide (Mg(OH)₂) was purchased from RMCC Company and maleic anhydride polyethylene were obtained from Banzan Co. Irganox 1010 as an initial antioxidant and Irganox 168 as a secondary oxidant were supplied from Nanjing Benjie Chemical Co. Zinc stearate (ZnSt) was purchased from Nanjing Lepuz Chemical Company. High density polyethylene (HDPE) (EX3-100S (HMCRP100N) and black masterbach were obtained from Maroon and Noavaran Baspar Company.

2.2 Preparation of compound

For preparation of the compound firstly 25 kg Maroon HDPE granules and 0.625 kg of black master bach granules was mixed properly and in the following stage, 42 kg Mg(OH)₂ as the flame retardant additive, and 2.11 kg maleic polyethylene for final modification was added. At the final stage 0.325 kg of any of the

following materials; Irganox 1010 as initial antioxidant and Irganox 168 as secondary antioxidant and ZnSt as lubricant was added to the mixture. For this experiment, the material was dry-blended by using a single extruder and then vacuum loaded into the extruder hopper. Although it is typical to perform drying step prior to pipe production therefore the blend was dried prior to pipe production. At the end, the resulted compound was analyzed [15-19].

2.3 Pipe Production

The system for manufacturing corrugated pipes includes two chillers with capacity of 20 and 30 ton, one vacuum device, two single screw extruders for each layer of pipe, two pressure sensors and twenty-two temperature sensors. This machine is equipped with twenty heaters, one electrical saw, six Zimens controllers for controlling temperature and length. This apparatus is manufactured by DROSSBACH Company in Germany with capacity of 1100 kilogram per hour (317 + 289 watt) and L/D 40.

2.4 Instruments

Thus the prepared compound, the pristine HDPE material, the prepared corrugated pipe and the pristine pipe were analyzed physically by OIT device to compare the oxygen induction time according to ISIRI 7186-6 Standard, however the DSC test was done for comparing the thermal behavior of mentioned materials. Melt flow index of materials and pipes was measured by MFI device in accordance with INSO 6980-1 standard. Density was measured through density kit and ash content was done by electrical furnace in the basis of ISIRI 7090-1 and ISIRI 6964 standards respectively. Ring stiffness test, flexibility and falling weight experiment was done according to ISIRI 11436, ISIRI 10607 and ISIRI 11438 standards respectively. The laboratory and field experiments are asserted in the following.

3. EXPERIMENTS IN THE LABORATORY SCALE

Two samples from corrugated pipes made from flame retardant compound and maroon granules respectively, was experimented in laboratory. After exposure of corrugated pipes with flame retardant material in direct flame, the mentioned pipe was extinguished quickly but the pipe without flame retardant materials became aflame rapidly and was destroyed completely (According to the figure 1).

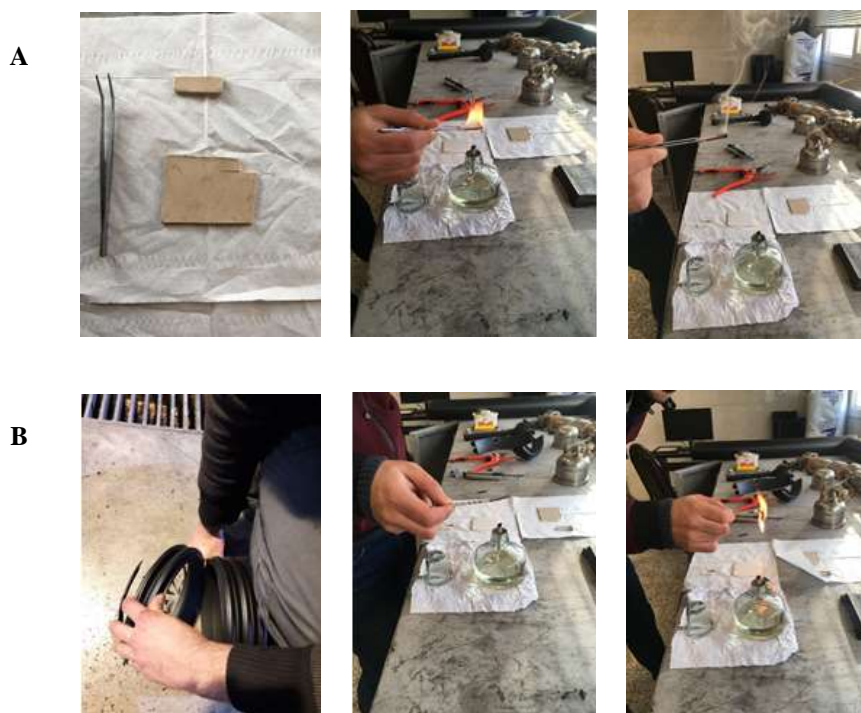


Fig. 1. Photograph of laboratory scale experiment

4. EXPERIMENTS IN THE FIELD SCALE



These experiments were conducted by exposing flame retardant corrugated pipes in direct fire for specific time period in 5 days at different directions (vertical, horizontal and at 45° angle) according to the table 1.

Table 1. Field scale experiments of flame retardant corrugated pipes

| Flame retardant corrugated pipe | | 20 sec | 30 sec | 40 sec | 50 sec | 60 sec |
|---------------------------------|------------|--------|--------------|----------------------|--|---|
| First experiment | Horizontal | ✓ | ✓ | Minor flame happened | Slight flame happened and distinguished spontaneously. The overall shape remained unchanged. | The pipe began afire but distinguished spontaneously. The overall shape remained unchanged. |
| | Vertical | ✓ | ✓ | ✓ | ✓ | ✓ |
| | 45 degree | ✓ | ✓ | Minor flame happened | Slight flame happened and distinguished spontaneously. The overall shape remained unchanged. | The pipe began afire but distinguished spontaneously. The overall shape remained unchanged. |
| Second experiment | Horizontal | ✓ | ✓ | Minor flame happened | Slight flame happened and distinguished spontaneously. The overall shape remained unchanged. | The pipe began afire but distinguished spontaneously. The overall shape changed slightly. |
| | Vertical | ✓ | ✓ | ✓ | ✓ | ✓ |
| | 45 degree | ✓ | ✓ | Minor flame happened | Slight flame happened and distinguished spontaneously. The overall shape remained unchanged. | The pipe began afire but distinguished spontaneously. The overall shape changed slightly. |
| Third experiment | Horizontal | ✓ | ✓ | Minor flame happened | Slight flame happened and distinguished spontaneously. The overall shape changed slightly. | The pipe began afire but distinguished spontaneously. The overall shape changed obviously. |
| | Vertical | ✓ | ✓ | ✓ | ✓ | ✓ |
| | 45 degree | ✓ | ✓ | Minor flame happened | Slight flame happened and distinguished spontaneously. The overall shape remained unchanged. | The pipe began afire but distinguished spontaneously. The overall shape changed slightly. |
| Fourth experiment | Horizontal | ✓ | Minor change | Minor flame happened | Slight flame happened and distinguished spontaneously. The overall shape changed slightly. | The pipe began afire but distinguished spontaneously. The overall shape changed obviously. |
| | Vertical | ✓ | ✓ | ✓ | ✓ | ✓ |
| | 45 degree | ✓ | Minor change | Minor flame happened | Slight flame happened and distinguished spontaneously. The overall shape changed slightly. | The pipe began afire but distinguished spontaneously. The overall shape changed obviously. |
| Fifth experiment | Horizontal | ✓ | Minor change | Minor flame happened | Slight flame happened and distinguished spontaneously. The overall shape changed slightly. | The pipe began afire but distinguished spontaneously. The outer layer of pipe destroyed. |
| | Vertical | ✓ | ✓ | ✓ | ✓ | ✓ |
| | 45 degree | ✓ | Minor change | Minor flame happened | Slight flame happened and distinguished spontaneously. The overall shape changed slightly. | The pipe began afire but distinguished spontaneously. The outer layer of pipe destroyed. |



Table 2. Field scale experiments of pristine corrugated pipes

| Pristine corrugated pipe | 20 sec | 30 sec | 40 sec | 50 sec | 60 sec |
|--------------------------|---|---|--------|--------|--------|
| Horizontal | The outer layer which was exposed to fire was completely destroyed. | The pipe began afire and the fire developed to all parts and the pipe destroyed completely. | - | - | - |
| Vertical | ✓ | ✓ | - | - | - |
| 45 degree | The part of pipe which was in the expose of direct fire destroyed completely. | The pipe began afire and the fire developed to all parts and the pipe destroyed completely. | - | - | - |

In the figure 2 sample placement has been shown after 60 seconds.

As it can be seen from table 1, the corrugated pipe made from flame retardant materials remained its shape stable after 3 times of flaming and after that just the outer layer will be destroyed. Another fact that can be understood is that until 30 seconds in consecutive days no changes was observed. Because of fire concentration in corrugated pipe, no change was occurred in the structure of the pipe.

Short term ring stiffness test, flexibility with 5% deformation and impact test using one kilogram weigh from 2 meter distance was done after and before the fire resistance test in the exposure of direct fire.

5. RESULTS and DISCUSSIONS

In order to identify thermal behavior of the prepared compound and the corrugated pipe, DSC and OIT experiments was performed. As it can be seen from figure 3, the prepared compound and its corrugated pipe have OIT duration of about 200.24 and 197.39 minutes respectively which is higher than accepted range according to 9116-3 ISIRI standard (OIT>20 min). Therefore it seems that the materials during the production process and application lifetime (according to standard) had not experienced oxidation degradation. However by comparing figure 3(a) and 4(b) it is understood that the oxidation time of pipes after adding flame retardant fillers increase significantly and this is due to the presence of mineral materials and their resistance against oxidation.

From MFI test, melt flow behavior has been identified. MFI results from raw materials and pipes are assumed in table 4. Melt Flow index of compound and the produced corrugated pipe is 0.7 and 0.76 in ten minutes at 190° C, respectively. The differential percent of MFI content is 12%, which is lower than the accepted value in 9116-3 ISIRI standard. The high value of melt flow index in flame retardant pipe and materials is because of presence of the mineral materials that retard the flame possibility.

The density values of corrugated pipes and the raw materials are given in table 4, as it is evident from the table, material density and its produced corrugated pipes density are 1.32 and 1.35, respectively which results in weight increase in comparison with other pipes. The reason underlying in the high density of these materials is the high percent of flame retardant mineral materials which were used during compounding [17, 18]. The final percentage of flame retardant mineral material and its corrugated pipe is determined by burning in electrical furnace at 900° C within 15 minutes. The results are presented in table 4.

DSC plot is shown in figure 4 and its relative data are assumed in table 5. Considering the high melt temperature of HDPE material comparing to flame retardant compound, the crystallinity value is notably higher and in consequence the produced pristine pipes have higher crystallinity toward flame retardant corrugated pipes, which suggests that addition of high amount mineral fillers decreases formation of crystals due to the chain motion limitation [20].

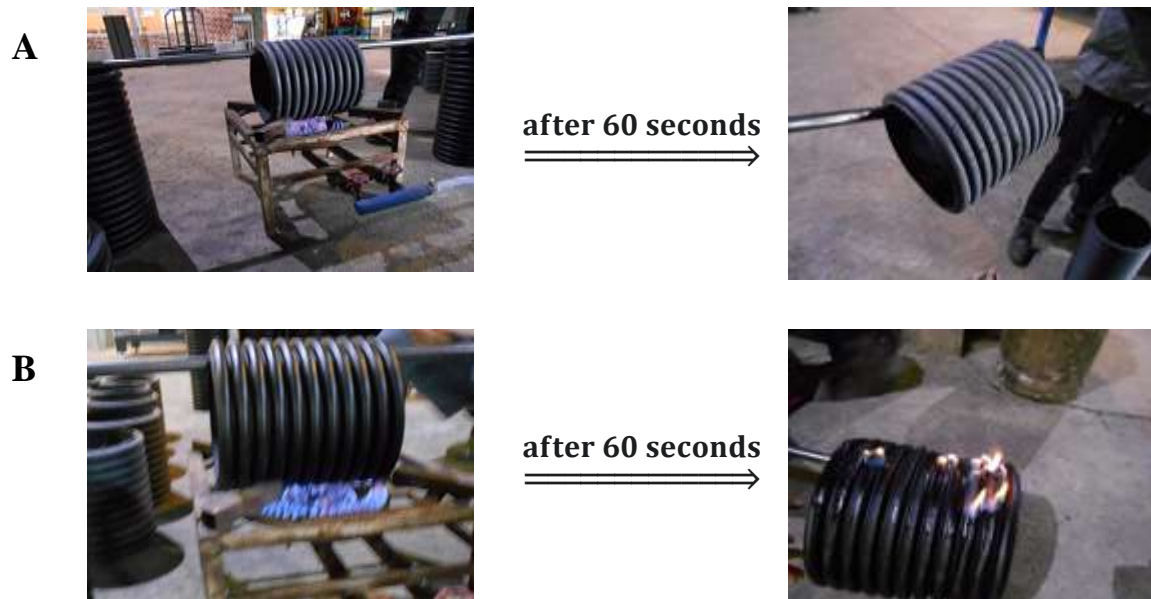


Fig. 2. Photograph of field scale experiment for (A) flame retardant corrugated pipe, (B) pristine corrugated pipe

Table 3. Short term ring stiffness test, flexibility with 5% deformation and impact test before and after fire resistance test

| Type of test | | Before flame resistance experiment | After flame resistance experiment (after 5 days) | | | | | |
|---|------------|------------------------------------|--|-------------------|-------------------|-------------------|-------------------|--|
| | | | | | | | | |
| Short term ring stiffness | | $16 \frac{kN}{m^2} \leq$ | $8 \frac{kN}{m^2} \leq$ | | | | | |
| Flexibility (5%) | | Without fracture | Without fracture | | | | | |
| Impact strength | | TIR \leq 10% | TIR \leq 10% | | | | | |
| Size change after flame test (Inner diameter before test=200.9) | Horizontal | - | 20 sec | 30 sec | 40 sec | 50 sec | 60 sec | |
| | | | 0.63% (199.62) | 1.72% (197.45) | 1.70% (197.50) | 1.97% (196.94) | 5.04% (190.77) | |
| | Vertical | | - | - | - | - | - | |
| | 45° | | 0.65% (199.60) | 1.63% (197.63) | 1.57% (197.74) | 2.49% (195.94) | 1.53% (203.97) | |

For evaluation of mechanical properties of corrugated pipes two experiments were applied, one is ring stiffness (strength against external pressure) with two modes (short-term & flexibility) by using ring stiffness device and the other is impact strength test by using a falling ball.

In figure 5(d), short-term ring stiffness of two different corrugated pipes are compared, as it can be observed short-term ring stiffness of flame retardant corrugated pipe is less than pristine corrugated pipe. Because of high percentage of flame retardant materials the value of short-term ring stiffness in recently made corrugated pipes is low which results in low strength in addition to flame retardant behavior. However according to figure 5(a), (b), (c), flexibility test was shown in 5, 10 and 15 percent of deformation respectively and it was evident that corrugated pipes made up from flame retardant materials show resistance toward shape change until 15% of deformation and in the following it fractured. The reason is high loaded mineral flame retardant materials in corrugated pipes which declines flexibility [16- 20].

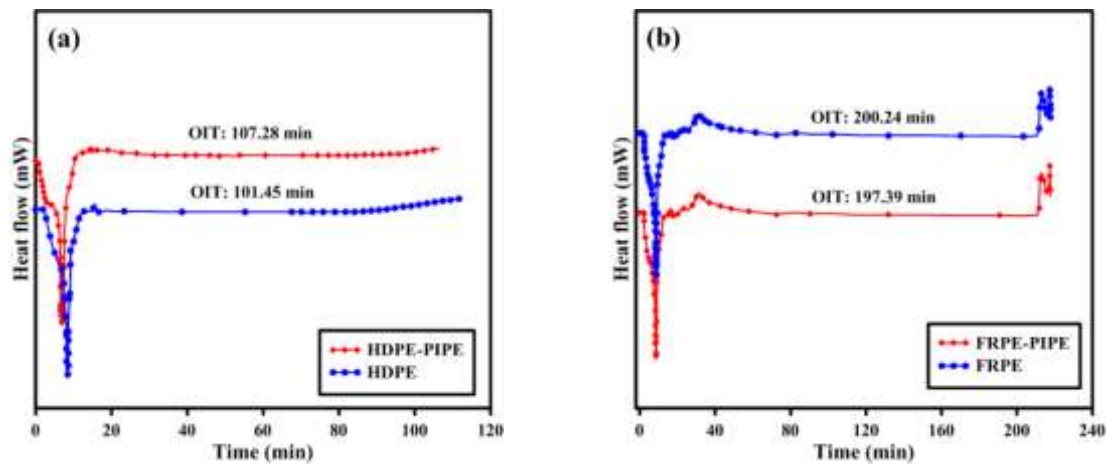


Fig. 3. OIT curves of material, compound and pipes ((a): pristine material and pipe, (b): flame retardant material and pipe)

Table 4. Density, MFI and ash content of compound, raw material, compound and pipes

| | HDPE compound granule with flame retardant materials | Produced corrugated pipe from HDPE compound | Maroon granule | Produced corrugated pipe from Maroon granule |
|------------------------------|--|---|----------------|--|
| DENSITY ($\frac{g}{cm^3}$) | 1.32 | 1.38 | 0.945 | 0.948 |
| MFI | 0.606 | 0.684 | 0.22 | 0.22 |
| Ash content | 37.43% | 36.62% | - | 2.2% |

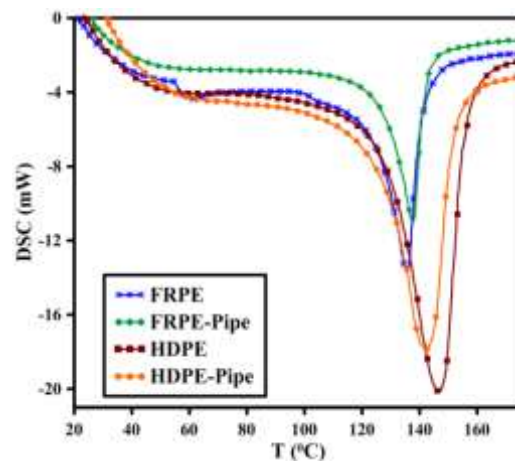


Fig. 4. DSC test of materials and corrugated pipes. (FRPE: flame retardant compound, FRPE-Pipe: flame retardant corrugated pipe, HDPE: Marron raw materials, HDPE-Pipe: pristine corrugated pipes)

The corrugated pipes resistance was determined by falling of 1 kg weight from the height of 2 meter. The experiment was continued until 25 impacts (according to ISIRI 11438 standard), in flame retardant corrugated pipes two impact led to crack formation and fracture while in pristine corrugated pipes no crack was observed. By far the TIR index for both types of pipes was lower than 10% which is acceptable in standard.

Table 5. DSC results of material, compound and pipes (FRPE: flame retardant material, FRPE-PIPE: flame retardant corrugated pipes, HDPE: maroon raw material, HDPE-PIPE: pristine corrugated pipe)

| Sample | T _m (°C) | ΔH (J/g) | Crystallinity (%) |
|-----------|---------------------|----------|-------------------|
| FRPE | 136.2 | 50.99 | 17.4 |
| FRPE-PIPE | 137.4 | 36.87 | 12.5 |
| HDPE | 147.1 | 104.88 | 35.7 |
| HDPE-Pipe | 142.25 | 89.31 | 30.4 |

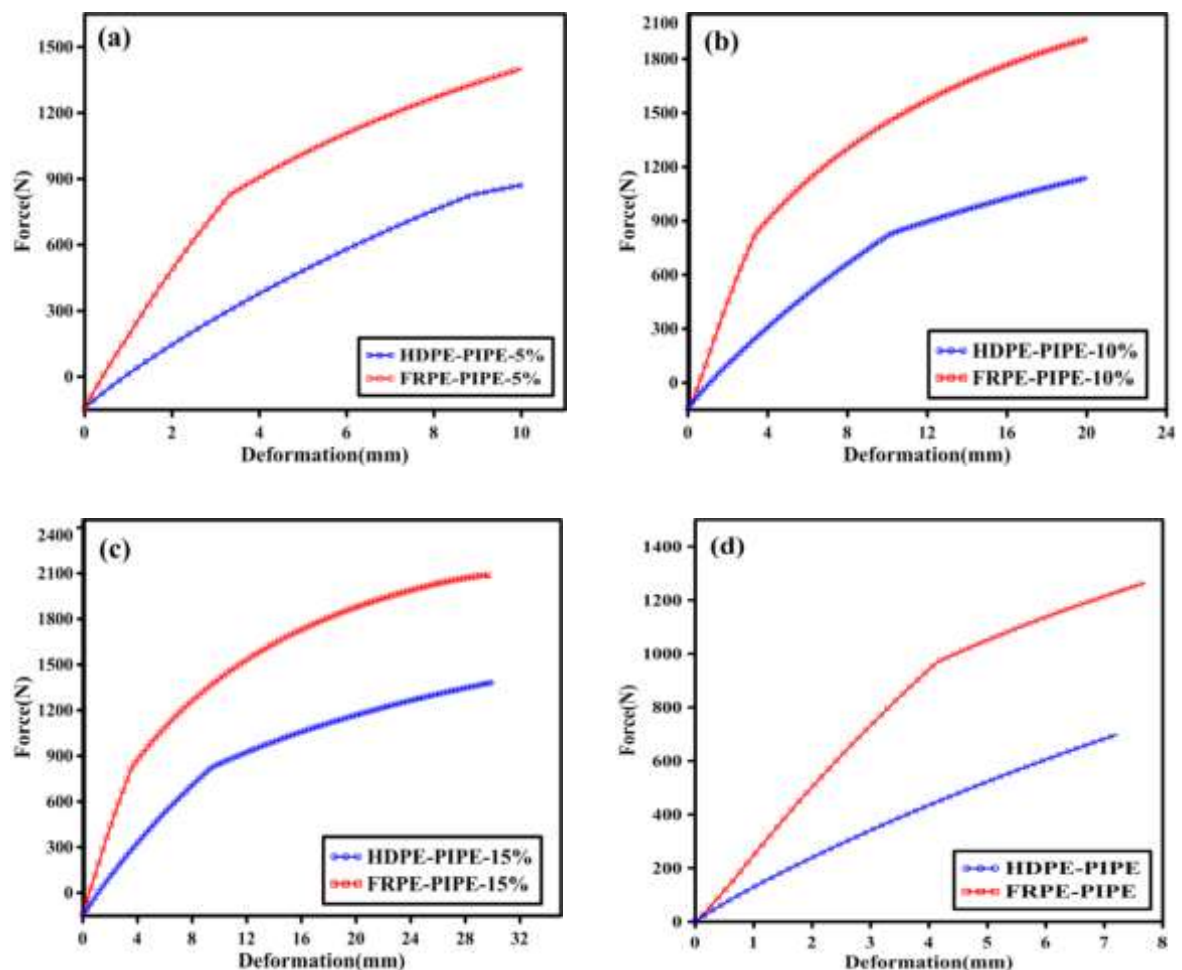


Fig. 5. Comparison of flexibility and short ring test of pristine corrugated pipes (HDPE) and flame retardant corrugated pipes (FRPE) at room temperature ((a): flexibility test at 5% deformation, (b): flexibility test at 10% deformation, (c) flexibility test at 15% deformation, (d): short ring test.)

6. CONCLUSION

In this experimental study the flame retardant corrugated pipes were prepared for agricultural purposes. First of all the compounded materials were obtained by presence of the mineral additive based on magnesium di-hydroxide and in the following the corrugated pipes were produced via a single screw extruder. For evaluation of physical properties, materials and produced pipes were experimented. It is apparent from the results the achieved consequences are in defined range of MFI standards for instance the melt flow index of compound and the resulted pipe is 0.684 and 0.606 g/10 min respectively. However in order to figuring out the mechanical operation of pipes, flexibility and ring stiffness and impact strength tests were implemented. Measurements presented that the flexibility and impact strength of flame retardant pipes is lower than pristine corrugates indeed the high percent of mineral materials in the produced pipes. The results showed that after 5 times exposing in direct fire for about 60 seconds, the outer layer of pipes was destroyed. As a result the



mentioned pipes have satisfying physical and mechanical properties for using as a riser protector in farming lands.

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