Report –

Quality assurance of raw materials

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Nomenclature

HDPE	High-density polyethylene
PP	Polypropylene
DSC	Differential scanning calorimetry
OOT	Oxidation onset temperature
OIT	Oxidation induction time

Forord

Det følgende er en teknisk rapport som undersøker i hvilken grad fiskekasser plukket langs Norske- og Svenskekysten egner seg til gjenbruk som takplater til Håpets Katedral, som skal ferdigstilles ila. våren 2021. Rapporten er skrevet på engelsk for å kunne nå et så stort publikum som mulig, men en kort oppsummering av prosjektet og hovedfunnene gis i tillegg her på norsk.

Det starter med innsamling av råmaterialene. Dette har hovedsakelig blitt gjort på dugnad der drøyt 1000 fiskekasser har blitt samlet inn fra strender, fiskemottak osv. All ære til alle de som har stått på for å få tilstrekkelig med materiale på plass, anslagvis rundt 4 tonn!

Fiskekassene ble så vasket og sortert, både ved Maritime Center Fredrikstad sine lokaler på Isegran og hos Biobe AS på Lisleby, før hver fraksjon (sortert på farge) ble kappet i biter og malt opp til et råmateriale som kunne brukes i sprøytestøpemaskinene hos Biobe.

For å bli kjent med materialet både fra et produksjonsmessig og et materialteknisk perspektiv ble det deretter kjørt en serie prøveprodukter. Hos Re-Turn ble det så gjennomført analyser av de ulike materialene, og tiltak iverksatt for å sikre så lang levetid som mulig. Hovedsakelig har dette bestått av å inkludere ulike tilsetningsstoffer for å forbedre materialene på de punktene analysene avdekket at det var nødvendig.

Det er i hovedsak fokusert på to forskjellige parametere, varmestabilitet og UV (lys) stabilitet. Varmestabiliteten viste seg å være minimal, og det er helt nødvendig med tilsetning av additiv, såkalte antioksidanter, for å sikre at materialene bedre tåler varmebelastningen de blir utsatt for (både under produksjon og i livsløpet). UV stabiliteten viste seg å være relativt god på den undersøke fraksjonen (naturell), men Re-Turn anbefaler fortsatt å inkludere noe additiv i form av en UV-stabilisator, med tanke på den store lysbelastningen materialene vil bli utsatt for når de er montert på taket.

Oppsummert er det vist at materialet egner seg godt til støpning av nye produkter, men at tilsetning av egenskapsforbedrende additiver er nødvendig for å sikre lang levetid (5-15 år). Jobben som er blitt gjort med innhenting, sortering og vasking har resultert i et rent materiale som egner seg godt for mange applikasjoner. Dette gjør det også til et attraktivt materiale i markedet.

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1 Introduction

Re-Turn AS has been tasked by Håpets Katedral AS, in collaboration with Biobe AS, to perform quality assurance testing of the recycled raw materials intended for production of the roof shingles for Hope Cathedral, which is currently under construction.

The target lifespan of these products is very long, preferably ten to fifteen years. Coupled with the fact that the products will be situated outside exposed to the elements and radiation from the sun, makes careful selection of materials and additives imperative to minimize premature failure. In this case typically discoloration, chalking, cracking and eventual breakage.

Besides making sure that the materials have satisfactory ductility (by sorting out the most severely degraded (brittle) fish crates), it was decided to focus mainly on two different parameters, namely thermal- and UV resistance. Select materials were tested "as is", and with additives, until satisfactory results had been achieved, and a standard formulation was then suggested for implementation in production.

2 Overview of investigated materials

2.1 Raw materials

2.1.1 Virgin materials

- **Sabic HDPE M80064:** Commodity virgin high-density polyethylene material supplied by Sabic. Injection moulding quality. Used as a comparative basis for the recyclates.
- **Sabic PP 576P:** Commodity virgin polypropylene material by Sabic. Injection moulding quality. Used as a comparative basis for the recyclates.

2.1.2 Recyclates

- **HK HDPE Fish crates (natural):** HDPE fish crates collected from Swedish and Norwegian beaches, then sorted and shredded into flakes by Håpets Katedral. Injection moulding quality. Multiple colour fractions will be used in production, but natural was chosen for this study. The other colour fractions are assumed to display similar properties.
- **HK PP Beach:** Virgin PP granulate collected from Norwegian beaches after an accident occurred where a large amount of material was spilled into the sea during transport. Extrusion quality. Collected and cleaned by Håpets Katedral.

2.1.3 Additives

- **CESA®- nox 4102:** Concentrate of antioxidants in a PE carrier. Provides thermal protection of the polymer by preventing oxidation during processing and service-life, which is the main cause of aging in polymers.
- **CESA®- nox PE 10880:** Old version of CESA®- nox 4102, no longer available. Included for comparison.
- **CESA®- light 7109:** Concentrate of light stabilizers in a PE carrier. Protects against photochemical degradation, making light sensitive polymers suitable for outdoor applications.

2.2 Sample overview

Table 2-1 gives and overview of the various material blends investigated in the project.

Material ref.	**SN-100-65-03-00**	**SN-100-65-03-01**	**SN-100-65-03-02**	**SN-100-65-03-03**	**SN-100-65-03-04**	**SN-100-65-03-05**	**SN-100-65-03-06**	**SN-100-65-03-07**
Virgin materials								
Sabic HDPE M80064	100%							
Sabic PP 576P							80%	
Recyclates								
HK HDPE Fish crates natural		100%	96%	96%	94%	96%		
HK PP Beach							20%	100%
Additives								
CESA®- nox 4102			2%	4%	6%			
CESA®- nox PE 10880						4%		
CESA®- light 7109			2%					
SUM	**100%**	**100%**	**100%**	**100%**	**100%**	**100%**	**100%**	**100%**

Table 2-1 Sample overview

3 Test methods

The following is a description of the various tests, standards and equipment used in the project.

3.1 Preparation of test specimens

Dogbones of type 1A were injection moulded by Biobe AS, using a tool made in accordance with ISO 527-2. The specimens needed for the various tests where then machined from these dogbones.

Figure 3-1 Test specimens - Dogbones

3.2 Thermal resistance – OOT and OIT

Thermal stability can, by DSC, be characterized in two different ways, oxidation onset temperature (OOT) and oxidation induction time (OIT). The former of these indicates at what temperature oxidation initiates at a constant heating rate starting at ambient temperature, whereas the latter indicates the time interval until oxidation initiates at some constant chosen temperature (e.g. 190 is commonly used for polypropylene).

3.2.1 Oxidation onset temperature

The oxidation onset temperature was determined by DSC using a Netzsch DSC 200 F3 Maya (shown in Figure 3-2), according to ISO 11357-6. After a short initialization period at 40°C, the specimens were heated at a rate of 20 K/min in a pure oxygen atmosphere until oxidation occurred.

Figure 3-2 DSC equipment

3.2.2 Oxidation induction time

Oxidation induction time was determined by DSC using the same equipment as described in Chapter 3.2.1 according to ISO 11357-6. The specimens were heated, at a rate of 10 K/min, to the target temperature of 200°C in a nitrogen atmosphere, followed by a short stabilization period. The atmosphere was then changed to pure oxygen, and the time interval until onset of exothermic oxidation of the material was measured, ref. Figure 3-3.

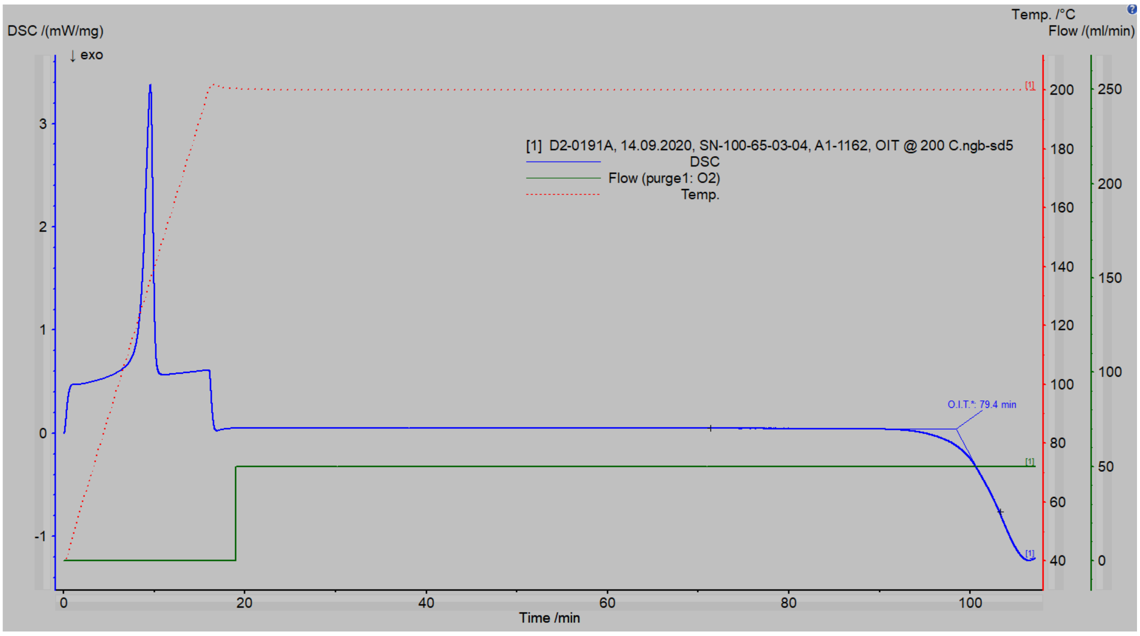

Figure 3-3 DSC OIT thermogram

3.3 Light (UV) resistance – Accelerated weathering

To simulate long term degradation of the materials due to exposure to the elements, a QUV/basic accelerated weathering tester was used (ref. Figure 3-4 to Figure 3-6). The test was conducted according to ISO standard 4892-3, method A, cycle 1, with the parameters shown in Table 3-1. UVA-340 lamps were used as their spectrum give the best available simulation of sunlight in the UV (295 to 365 nm) region, see figure 2-4, where most of the damage to materials occurs. The test was run for 1344 h (2 months) and one set of specimens was removed at the halfway point to investigate the incremental degradation of the materials.

Figure 3-4 Weathering equipment – Test setup

Figure 3-5 Weathering equipment – Test setup (2)

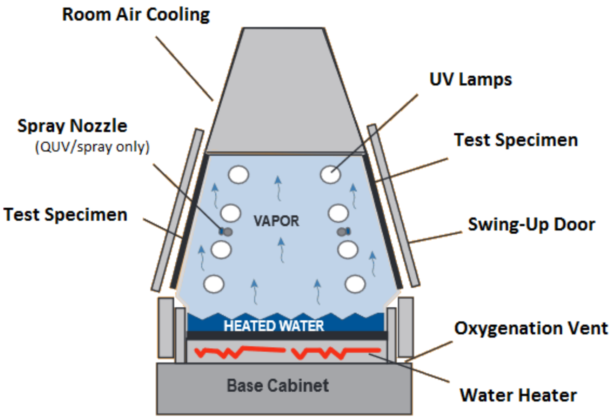

Figure 3-6 Cross section of the QUV

Cycle No.	Exposure period	Lamp type	Irradiance	Black-standard temp.	Relative humidity (%)
1	8 h dry	Type 1A (UVA-340)	0,76 $Wm^{-2}nm^{-1}$ at 340 nm*	60 °C ± 3 °C	Not controlled
	4 h condensation		Light off	50 °C ± 3 °C	

Table 3-1 QUV test parameters

*The irradiance cannot be controlled directly in the QUV/basic, the stated irradiance is the approximate average over the lifetime of the lamps.

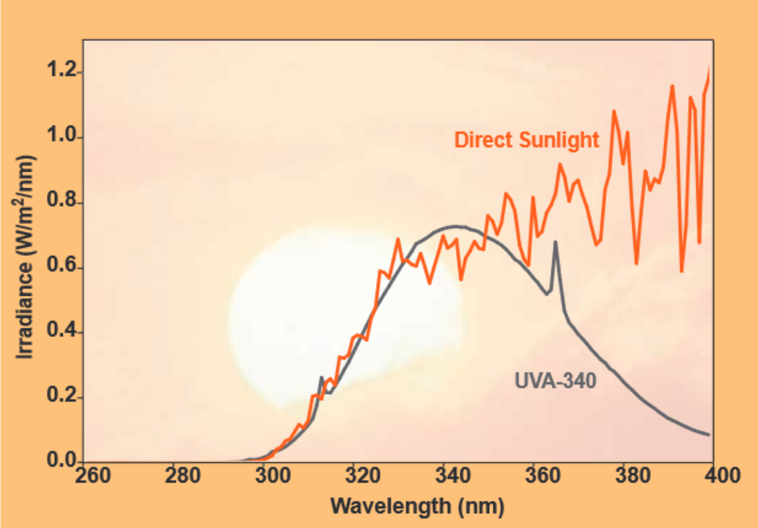

Figure 3-7 Lamp spectra

3.4 Impact Strength

Charpy impact tests were conducted using impact testing equipment from Alarge laboratory technologies (shown in Figure 3-8 below), in accordance with ISO standard 179-1. Type 1 specimens, machined from the dogbones described in chapter 3.1 were tested in the edgewise configuration, with a type A notch.

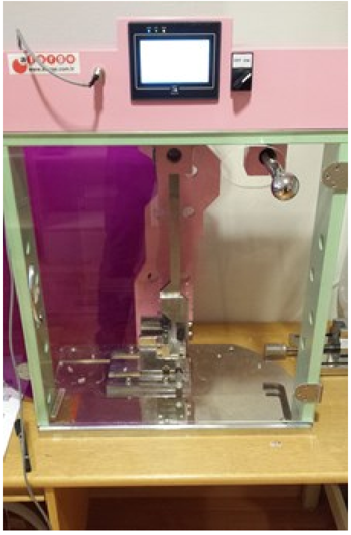

Figure 3-8 Impact tester from Alarge

4 Results

4.1 Heat resistance

The thermal stability of the various HDPE materials are shown in Figure 4-1 and Figure 4-2. Here, the thermal resistance of the pure materials (0 and 1), but also the material with 2% thermal stabilizer (2), is very low. Only at 4% or more a significant improvement in performance can be observed (material 3, 4 and 5).

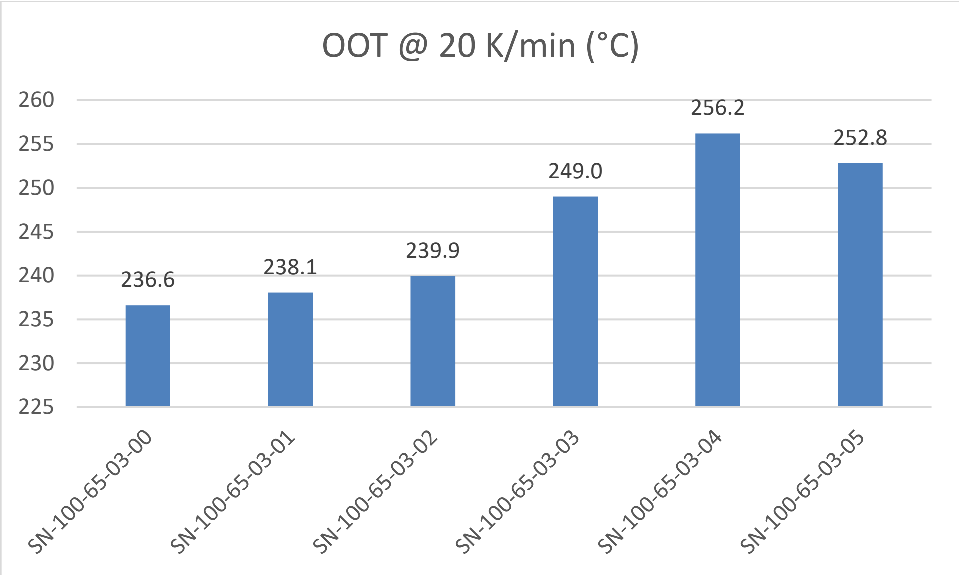

Figure 4-1 Results – OOT

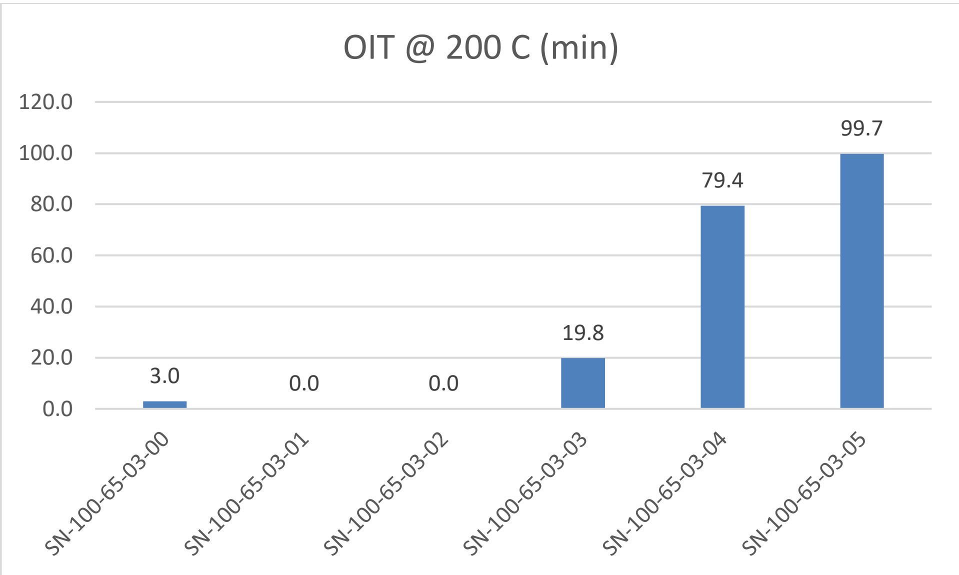

Figure 4-2 Results – OIT

4.2 Light (UV) resistance

UV testing was conducted on the following materials:

- SN-100-65-03-01: 100% HK HDPE Fish crates (natural)
- SN-100-65-03-02: 96% HK HDPE Fish crates (natural), 2% CESA-nox 4102, 2% CESA-light 7109
- SN-100-65-03-07: 100% HK PP Beach

The samples were then investigated for degradation by visual inspection, and by conducting Charpy impact testing.

4.2.1 Visual inspection

Figure 4-3 shows the various samples before starting the test. As can be seen in the picture, two sets of samples are present as one set will be removed from the machine after four weeks of exposure, while the second set was left until the end of the test (8 weeks).

Figure 4-3 Results – UV samples before exposure

Figure 4-4 shows the samples after exposure. From left to right material 1, 2 and 7 is displayed with one specimen for each exposure, cero, four and eight weeks, respectively. Of these, materials 1 and 2 show little sign of aging, even after 8 weeks. This is not the case for material 7 however, which shows significant chalking and cracking, already after just 4 weeks.

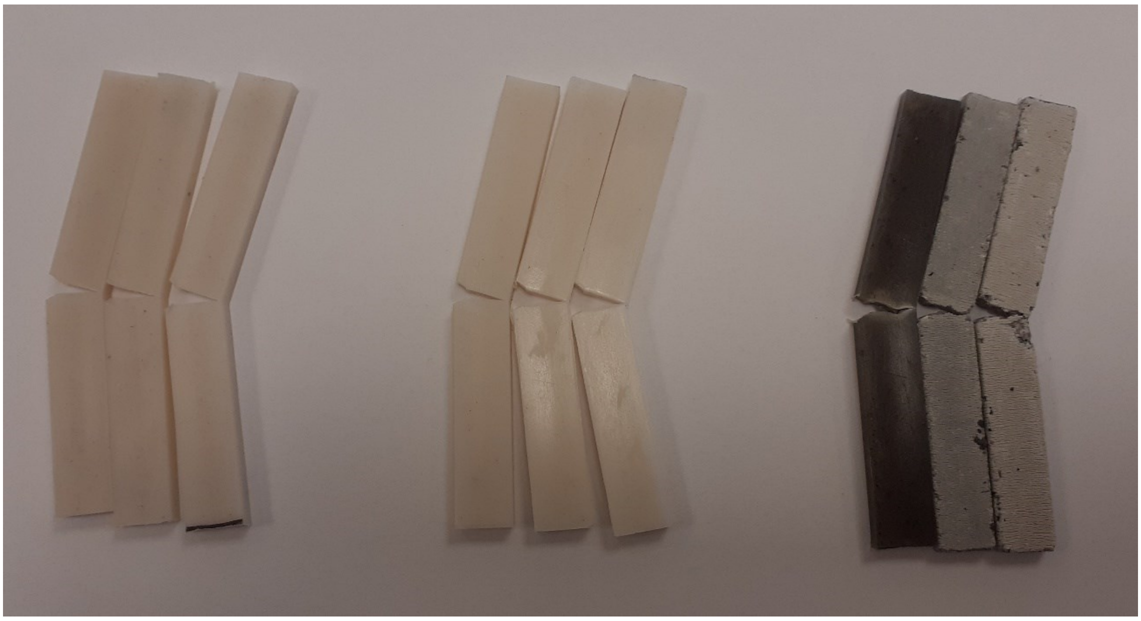

Figure 4-4 Results – Visual inspection after exposure

4.2.2 Impact resistance

The impact strength of the materials shows a similar tendency as for the visual inspection. Material 1 and 2 show only minor variation between each exposure period, whereas 7 show a clear reduction in strength between each exposure.

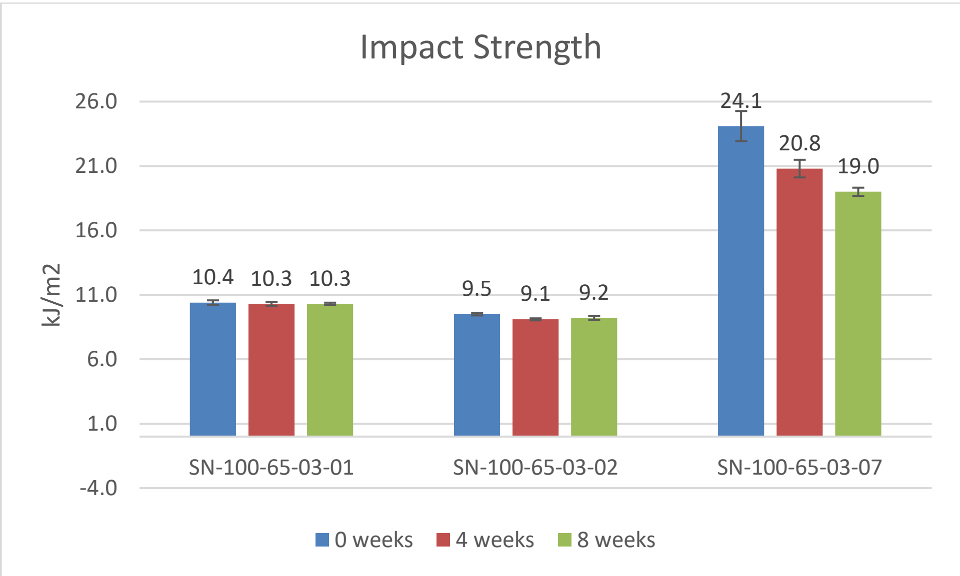

Figure 4-5 Results – Charpy Impact Strength

5 Discussion

5.1 Heat resistance

As is evident by the results shown in chapter 3.2, the thermal resistance of the fish crates recyclate is inadequate, and addition of thermal stabilizers (aka. antioxidants) is necessary to make it viable in new products.

Surprisingly, this also holds true for the virgin material, which also have a very low OIT of only 3 minutes. While the virgin material has not been the focus of this study, and will not be covered further here, Biobe has been informed so that the necessary measures can be taken should it be used in the future. It is worth mentioning that this seems to be a general trend, where the material properties are compromised as raw material producers reduce costly additives to the bare minimum to stay competitive in a very cost-driven market.

Of the three remaining materials, number 3 (containing 4% CESA-nox 4102) is considered the optimal choice, as it represents a good compromise between performance and price.

CESA-nox PE 10880 is regrettably no longer available in the market, but it was included here for comparison with 4102 as it (4102) is sold as an equivalent product. This is clearly not the case as we see a significant deviation between the two systems. This has been brought up with the supplier, but no response has so far been received.

5.2 UV resistance

As is evident by Figure 4-4 and Figure 4-5, the two HD-PE materials show no signs of degradation even after eight weeks (the variance is so small it's negligible). While this holds true even for the unmodified system, Biobe and Re-Turn still recommend adding UV stabilizers due to the harsh conditions the products will be exposed to.

It is obvious however that the additives slightly reduce the impact resistance of the materials somewhat. This could both be due to the additives themselves having lower impact strength, or it could be due to a slight incompatibility between them and the recyclate, but this could not be studied further within the scope of this project and should be considered a "necessary evil" to ensure satisfactory performance of the other properties.

As for the PP material, it is clearly not suited for use "as is". This could perhaps be expected, as the material has seen severe UV exposure by being exposed to the elements for several weeks as individual granules. Due to this fact a new set of samples have been moulded (SN-100-65-03-06) in an 80/20 mix of virgin and recyclate. Further, a new UV test has been scheduled for start in week 40, and the results will be shared as soon as they become available. Compared to the HDPE materials it does show significantly higher impact strength. This is expected though as this is an extrusion quality material, intended for pipe production, which typically have high impact strengths.

6 Conclusion

It has been shown that thermal stabilizers are necessary to provide a viable raw material for production, and that 4% of CESA-nox 4102 should yield the required performance. While the pure HDPE recyclate performs well in the UV test, addition of UV stabilizer is still recommended at 2% loading to ensure long-term UV resistance. Further, production of roof shingles in pure recycled PP should be avoided as they will degrade very rapidly. A follow-up test has been scheduled to evaluate the viability of an 80/20 mix of virgin and recyclate, with results being shared as soon as possible.

6.1 Formulation suggested for production

The following material is suggested:

- HK HDPE Recyclate
- 4% CESA-nox 4102
- 2% CESA-light 7109

Colour masterbatches can be added to this base as needed.